

Emil/Laurence Smith

INVESTIGATE ZERO-STRESS REPLICATED OPTICS

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Darell Engelhaupt

**The University of Alabama in Huntsville
Center for Applied Optics
Huntsville, Alabama**

Robert Rood

**NASA Marshall Space Flight Center
Optical Fabrication Division
Huntsville, Alabama**

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AXAF-S DEVELOPMENT COMPONENTS NAS8-38609 D.O. 63

THE UNIVERSITY OF ALABAMA IN HUNTSVILLE

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To: Angela Fizah (UAH Contracts) (205) 895-6000
R. Rood NASA MSFC, Dr. John Dimmock UAH

From: D. Engelhaupt

Subject: UAH Final Report

CC: J. Bilbro, C. Jones, EB23, P. Monefo, EH24
Distribution per R. Rood

Robert Rood,

The contracted activities for the procurement of "Investigate Zero-Stress Replicated Optics" to support the AXAF-S x-ray spectrometer mirrors has been completed. To date four large Wolter I grazing incidence x-ray optical shells have been electroformed from nickel. The mirrors were fabricated utilizing each of two nickel alloy plated aluminum substrates twice. A wide variety of testing has been completed by NASA MSFC and UAH. This testing includes heat treatment control tests, subscale plating and fixture testing, alloy control of the electroless nickel, adhesion and release testing of the gold to electroless nickel, electroforming instrumentation and software and fabrication of subscale models. The full scale shells are one millimeter thick nickel electrodeposited over a thin gold layer which in turn has the optical surface on the inside. The optical surface is the replicate of the surface prepared on the substrate. Appendix I briefly outlines the fabrication process.

Major objectives which were shared by UAH and MSFC include the design of facilities, equipment and tooling and procurement of materials and equipment. Process development followed with the fabrication of small scale pilot units. Procurement commenced immediately and equipment and materials were ordered to implement the fabrication of first surface full scale substrates (mandrels) and the second surface electroformed optical components.

All principal objectives have been achieved. Inspection of the mirrors in visible and x-ray modes validates that the required performance and the quality can be achieved by an electroforming replication process. A very distinct progressive improvement has been achieved with each of the four mirrors produced. The final mirror exceeded the original goals and set an improved standard for flight hardware. The future goal of a 30 arc second resolution at 8 KEV x-ray appears to be achievable by this process when proper cleanliness and process control is utilized.

ACTIVITY:

1.0 Laboratory and Facilities Efforts Supported by UAH:

UAH consulted with NASA Facilities personnel to expedite installation of the laboratory utilities and equipment needed for the plating of the large parts. This includes running 220 volt two phase and 208 three phase lines, removing the existing water lines and installing the two deionized water lines. One of the water systems is about two megohm-cm purity and the other is about twelve to eighteen megohm-cm. The lower purity water is continuous fed and may be used to rinse parts and the higher purity water which is limited supply, is to be used for all steps when the supply is adequate and always to make up the solutions. An overhead hoist was installed including the required electrical wiring. A weir has been built to contain any possible accidental spill of plating chemicals. The ventilation system has been completely revamped including installation of external ducts to remove fumes and installation of a very large bag filter system.

The plating tanks were received and installed on schedule. The electroless nickel system was delivered by the manufacturer on time. They drove the equipment in from central Florida to assure expedient delivery. All of the chemicals were received on schedule. An estimated 20 troy ounces of various gold processes were sent in for reclamation. This was applied to the cost of 35 gallons of gold solution.

2.0 Mandrel Fabrication:

2.1 Aluminum Alloy Selection:

The selection of 2024 aluminum alloy was the first step towards fabrication of the mirrors. This alloy was chosen since it can be forged and near-net forgings were commercially available. Also the copper content of about 4.3% and low zinc permit plating substrates to be produced more easily than for high strength alloys such as the 7000 series. The high copper alloys are less subject to plating defects in the form of pits due to inclusion of foreign materials such as iron and silicon oxide (glass) chips found in 6061 alloy. An optional aluminum alloy choice would be 2219 at 5.3% +/- 0.5% copper.

The 2024 aluminum cylinders were received in the 2024-F condition which simply means "as forged". This designation was clarified by the vendor as follows:

- 1) 21 hours at 850 deg. F.
- 2) Forge until temperature drops to 700-740 deg. F.
- 3) Reheat at 850 deg. F. for 3 hours.
- 4) Second swage with temperature above 700 deg. F.
- 5) Air cool and Ship

This temperature is below the solution heat treatment temperature of 910-930 deg. F for 2024 to achieve the T4 or T42 condition. Since no quench was applied, the internal stress will be primarily related to the second forging operation and will consist most likely of external tensile stress and internal compressive stress consistent with shaping the part. Therefore the entire heat treatment sequence was specified. The quench hardening specified was designed to avoid the introduction of residual stress and the aging was selected to completely relax the material to improve the optical stability.

2.2 Aluminum 2024 Alloy Heat Treatment Specification:

The specification of a heat treatment schedule was made for producing a very low residual stress machined mandrel from the swaged forgings of 2024 Al in the "F" condition. Since there was no precedence available for this exact task it was decided the best approach would be to overage the first mandrel material to eliminate all residual stress in the material. Subsequent NASA tests showed that it did harden and then was annealed to a lower hardness as a result of the post solution treatment which was to stress relieve any machining induced stress. The temperature and times were altered slightly for the second mandrel and it was slightly harder as explained later. Actual performance of both mandrels in single point diamond turning was very good.

Specifications and calculation methods for cutting circular rings from the forged shapes and observing the change in a gap cut through the ring were made by UAH. Very little residual stress was determined to be present in the alloy forgings by this method.

NASA MSFC chose to study this in more detail and subsequently reduced the overaging time and temperature to improve the hardness and render the second mandrel more easily machined. The recommendation was to reduce the temperature of the annealing step from 500 deg. F. to perhaps 400 deg. F. Also the solution soak time was reduced from 18 hours to 12 hours in order to preserve additional hardness. This provided additional hardness and according to the tests performed did not increase the residual stress in the aluminum which appeared to be very low in all the tests. Only a slight improvement in hardness was achieved but some possibility of the manufacturer having overheated the forging was noted in that the grain boundaries of the material had significant precipitated copper, a condition which is not reversible. Thus the softer material was processed successfully both times independent of the slight changes in the process. Data from this test is contained in the NASA MSFC report by Julian Bynam, EH24 "Investigation of AXAF-S 2024 Aluminum Forging Heat Treatment Response".

2.3 Heat Treatment Summary:

Material purchased in the T-6 condition is hardened and in thick shapes will contain stored energy in the form of non-uniform internal stress. This stress will cause deformation of the parts in machining but should not cause surface changes.

Subsequent thermal stress relief will relax internal stress after machining and no serious changes will occur to the surfaces at temperatures less than 500 deg. F (actually some alloys can sustain 650 deg. F but the definition of "No Damage" is unclear). The higher the temperature and longer the time of stress relief the more relaxed the component will become at the sacrifice of yield strength and ultimate strength. The elastic modulus remain essentially constant throughout all heat treatment cycles. Therefore the elastic stiffness is the same even though the yield and ultimate strength values change.

If the part is fully annealed (650-750 deg. F) it is nearly back to the solution quenched condition in as much as the solution of the copper but the pinning sites are farther apart and the grain structure may suffer some growth which can ultimately affect the surface. Forging at this point would have introduced severe stress again.

Returning to the solution heat treatment permits the quenching and subsequent liquid nitrogen/steam shock cycle described to be utilized for lowering the internal stress prior to precipitation hardening. It is imperative that the temperature be controlled to avoid the chance of eutectic melting of aluminum-copper and other trace eutectics which would require a complete remelt of the metal to alleviate.

An alternate similar cycle could be used, i.e. quenching from an annealing temperature and cycling through the extreme temperature shock then precipitation hardening, with no detrimental effects except that no data exists for the exact amount of residual stress expected. Extreme thermal cycles introduced without returning to the solution condition will reduce stress which can be relaxed by grain boundary motion such as cross slip. This may include relaxation of stress caused by other metallurgical processes by allowing an equilibrium to be attained. This cycle should not be introduced after diamond turning the optical figure in the aluminum nor after the final diamond turning of the nickel mirror surface to avoid the chance of minute changes affecting the optical figure.

X-ray stress analysis was not used since the capability did not exist at UAH or NASA MSFC. This process is also limited analytically to about 3000 psi. Positive or negative values can be determined in this range. However the stress measurements are only taken at the surface and the stress may be distributed in

unknown ways throughout a machined and heat treated component. For plates the stress may be determined by relaxation measurements of a drilled hole. This will be difficult for the heavy cylindrical shape. Also the schedule requirements mandated continued processing.

For the AXAF-S application it is not mandatory or even desirable to achieve the highest hardness or strength values. The objective is to reduce the tendency to age by continued metallurgical changes due to extended time or processing steps. At least two cases exist wherein this could occur:

- 1) During the electroless nickel plating after all aluminum machining is complete, temperatures of about 180-190 Deg. F for as much as 40 hours depending on plating rate and thickness requirements.
- 2) During post plating heat-treatment for adhesion of the electroless nickel-phosphorous alloy.

Additionally if the material is left in the stressed forging condition with no solution heat treatment it will age spontaneously, microscopically deforming even at room temperature for up to one year. Additional deformation due to creep is possible at the present temper due to the horizontal cantilever machining if the part were left stationary for extended periods. Therefore the appropriate process is somewhat different due to requirements to achieve stable rather than ultimate properties.

Stress from diamond machining and nickel plating are minimal. No extreme thermal treatment should be introduced after diamond machining. The last fabrication step for the mandrel must be the final electroless nickel machining and polishing which is performed after the mild heat treatment to assure good adhesion.

See Appendix II for more detail on heat treatment of the aluminum and Appendix III for plating details.

3.0 Electroless Nickel Plating:

Two AXAF-S mandrels were processed through electroless nickel plating. The plating process selected was a complete commercially available activation and plating procedure reported to yield very sound deposits with the pitting and porosity near zero for sound substrates. The process was selected both for quality and for the absence of cyanide containing compounds which would have been hazardous to handle in the present laboratory.

The selected process includes all cleaning, oxide removal and strike coating steps including substitution of a nickel glycolic acid base strike which replaces a more conventional copper cyanide strike in many other processes. The adhesion values

reported appeared to be higher than that actually achieved as evidenced by some flaking at the edges of the electroless nickel when the parts were cut later. Concern with fixturing leaks which allowed carry-over of solutions is believed to be at least partially responsible and were addressed.

It was believed that the plating rate fell from about 0.25 to 0.30 mils/hour to about 0.15 mils/hour after the first part was plated which suggested contamination or that the temperature was too low. This was believed to cause the deposit thickness of the first mandrel to be only about 0.003 instead of 0.004 inches as predicted for the first mandrel. Later tests showed the plating rate falls spontaneously throughout the plating run even in new solution and at the highest recommended temperature. Most of the decline occurs in the first two hours and is due to the reduced catalytic activity of the nickel phosphorous alloy deposit compared to the pure nickel from the initial strike. The second mandrel was processed similarly but for 40 hours to yield a thickness of about 0.0045 inches.

4.0 Gold Adhesion and Separation Tests:

4.1 Test Samples:

The adhesion of the gold layer on the electroless nickel phosphorous is perhaps the most critical step. The gold must adhere sufficiently to prohibit any release during subsequent operations but must readily release after forming the nickel shell. Differential contraction of the shell and the aluminum mandrel is used to separate the final part. Liquid nitrogen is used inside the aluminum part to produce the temperature difference and contraction of the mandrel from the shell.

Adhesion test samples were first processed using a combination of sodium hydroxide and sodium dichromate to form a passive film on the electroless nickel-phosphorous alloy. The nickel was plated in a test set-up using the exact process to be used for the mirrors. The quality of both the nickel alloy and the low adherence gold was very good in most cases but persisted in non-uniformity of results. The use of the dichromate appears to be unnecessary in addition to unpredictable. Experiments to eliminate this step were conducted. This material is not only toxic but also a very serious poison to other related plating processes including the gold. Additional experiments were conducted using electrochemical polarization methods prior to plating the actual mirror and are described under the section on alkaline gold plating.

4.2 Acid Cyanide Gold Plating:

Brief tests plating from an acid gold cyanide solution onto nickel-phosphorous alloy of 6% phosphorous were inconclusive. By electrolyzing the nickel sample in an alkaline cleaner and then

passivating in a five gram/liter sodium chromate solution it was possible to achieve various levels of adhesion. Using cathodic treatment followed by simple immersion in the chromate solution no release of the gold was observed. By making the sample cathodic in the cleaner for 30 seconds and then anodic in the chromate solution, complete passivation occurred and the highly stressed acid gold cyanide process failed to produce an adherent deposit causing blistering and peeling of the gold.

4.3 Alkaline Sulfite Gold Plating:

Subsequent polished one inch diameter flat samples of 11% phosphorous NiP deposit were gold plated in a Technic 25 (TM) alkaline gold sulfite process.

The initial passivation consisted of immersion in 5 grams/liter NaOH for 30 seconds and 5 grams/liter K_2CrO_3 for 5 seconds without current. A smooth gold deposit was obtained which readily lifted with cellophane tape. Deposition of nickel onto several of these samples resulted in good separation. However the results were not readily repeated. Additional tests were performed using electroless nickel-phosphorous of 11% phosphorous plated on conical samples. The passivation steps were completed and gold from the alkaline process was applied followed by a heavy electrolytic nickel plated coating which simulated the entire process. These samples were then pressed on a universal load tester and the value of adhesion measured directly. The adhesion appeared to follow one of two paths and had about 800 to 2000 pound per square adhesion or about only 5 to 10 psi depending on the nature of the passivation test. It was determined that at the low level of adhesion cellophane tape would readily lift the gold from the electroless nickel and that if the adhesion was not satisfactory the tape would not release the gold.

Tape pull release of the gold was readily achieved on samples without the use of the dichromate solution. Passivation at low anodic current from 2 to 4 mA/cm² from 5 to 10 seconds resulted in film formation which could be gold plated and subsequently the gold could be lifted with cellophane tape. When samples were plated using 5 or more mA/cm² for 10 seconds anodic passivation in 5 gm/liter NaOH the gold was not uniformly deposited and would often wash off with running water.

Similarly a test Ni/Au shell plated on a substrate made cathodic in 5% NaOH at 18 mA/cm² for 60 seconds and then permitted to soak for 30 additional seconds did not readily release when subjected to the LN₂ immersion. This sample had exposed aluminum which was attacked by the NaOH solution. This in turn would force the nickel phosphorous plated portion to remain cathodic due to galvanic corrosion potential differences. This is believed to be at least partially responsible for the gold adherence to the nickel-phosphorous.

In order to gain better control of the adhesion a more fundamental understanding of the nature and behavior of oxide films on high phosphorous nickel alloys was required.

The formation of oxide films on alloys proceeds differently depending upon many conditions. First, spontaneous oxide formation in air or water occurs for most metals including nickel. The time and temperature determine the extent of oxidation. The film formed will generally increase in oxygen toward the surface. The oxide film is generally semiconductive with local sites of higher conductance as a result of film formation defects. The "defect" sites have been studied for many systems and agreement is that increased ionic and electron mobility occurs when the atomic arrangement surrounding atoms of reduced coordination is not of the same stoichiometry as the bulk film. This generally causes patches of non-conductive oxides in a field of conductive points. For pure nickel the progression of oxide formation is in the order; Ni(OH)_2 , Ni_3O_4 , Ni_2O_3 and NiO_2 . A similar progression is evident for the 11% phosphorous alloy also except that the phosphorous is present in the film.

Although tests are not complete at this time, the formation of the same number of stoichiometric layers is evident. At this time it is believed that the OH layer is actually also a phosphorous rich layer in the absence of substances with which phosphorous can form complexes. This layer formed in sodium hydroxide alone may therefore be $\text{Ni(HPO}_4\text{)}$ or perhaps $\text{Ni}_2(\text{HPO}_4)\text{OH}$ containing soluble diorthophosphate ions and proceeding to a solid substance immediately. The next stage of oxide formation would be expected to contain an insoluble or at least low solubility phosphorous compound such as P_4O followed by a higher oxidation number substance in combination with nickel such as P_2O_4 or P_2O_5 . The exact final product is not clear due to the similarity to pure nickel in the voltammograms.

By adding a complexing agent such as tartrate, the progression of the oxide formation is reduced to two observable steps (i.e., slow) and a more negative and stable reduction of the oxide is observed.

Ongoing electron spectroscopy chemical analysis (ESCA) and energy dispersive x-ray (EDX) analysis verifies that the phosphorous is indeed included in the oxide film. The data to date indicates that higher levels of oxide are formed in the solution of sodium hydroxide and no complexing agent. Also an initially high level of carbon is found. This is believed to be either from the atmosphere or from CO_2 absorbed by the alkaline solutions. Carbon is present in higher amounts on the sample oxidized in just sodium hydroxide indicating that oxidation of the tartrate is not responsible.

When gold was deposited on very passive NiP surfaces such as obtained in strong anodic passivation in NaOH or CrO_3 , the gold appeared brown and poorly adherent. The discolored gold deposits are most likely dendritic spires of gold occurring at the defect sites. Adherence will occur at these sites and the gold will not plate smoothly across the non-conductive patches due to localized high current density at the "defect" sites.

The acid cyanide gold-cobalt process first employed would reduce thin oxides due to complexation with cyanide and the evolution of hydrogen concurrent with the gold deposition in this process causing a high adhesion even if a thin oxide passivation layer is present. The alkaline sulfite gold process will not readily reduce the oxide film thus permitting deposition with low adherence on the passivated NiP surface permitting separation of the shell later. If the concentration of the gold is reduced below the minimum of one troy ounce per gallon recommended by the manufacturer the deposit may become diffusion limited and some hydrogen evolution will occur. This causes partial reduction of the oxide and patchy adhesion based on local differences in the current density and agitation.

The need is therefore to achieve a very thin and uniformly conductive oxide (semiconductive) film with no defect sites or with such a multitude of defect sites that the conduction appears to be continuous. After forming a very thin oxide film and upon immersion in a hexavalent chromium solution a semiconductive film (perhaps II-VI in nature) is formed and permits uniform deposition with the required low adhesion.

Since chromium is both toxic and a substantial contaminant for other plating operations it is desirable to eliminate this step. Also the 5 second immersion time found suitable on the small samples is not achievable on the actual AXAF-S hardware due to the size and weight of the mandrel and the plating fixture which requires a slow hoist to perform the maneuvering. An anodically formed film with an included cation is believed to be most appropriate. The substitution of nickel for the chromium would be desirable. Thus alkaline processes containing nickel complexes were considered. If the nickel-phosphorous alloy is made anodic in an alkaline nickel tartrate it is possible to form a uniform conductive nickel rich oxide surface rather than a mixed nickel oxide, phosphorous patchwork surface.

4.4 Vacuum Deposited Gold:

Vacuum deposition of pure gold on a clean Ni-11%P substrate produced adherence over most of the part but locally adherent areas were occasionally difficult to release. This resulted in small patches of gold not releasing from the mandrel. Deposition of gold in a vacuum system was performed both on test parts and on the last full size part. The parts had to be rotated in the

vacuum system to achieve deposit uniformity. The large part was vacuum gold plated at VERO in Dallas, TX using their existing rotational equipment. There was no forced passivation applied to the components which were plated by sputtering of the gold. Tests of surfaces which were not electrochemically passivated showed that the nickel oxide film was about the same depth as those which were electrochemically passivated. Interferograms also showed that the surface was not degraded by air passivation or by the electrochemical methods employed.

5.0 Low-Stress Nickel Plating:

The electrolytic low-stress nickel sulfamate plating process was implemented and additional studies performed to achieve uniform current density. This was done by measuring the field current at different input current levels using the actual mandrel. This was done via a commercially available instrument. In this way the anode position could be adjusted to compensate for non-uniformity in current density caused by the solution resistance and the second order field effects due to the mass transport phenomena associated with agitation and the migration of the nickel to the surface. A very small amount (diffusion limited) of an additive to cause compressive stress due to incorporation in the nickel grain boundaries as nickel sulfide was added to maintain the zero stress condition at a reasonable current density. This additive is 1,3,6, naphthalene tri-sulfonic acid, NTSA. Thus when the current was increased the ratio of the diffusion limited material would increase in nickel causing the stress to increase in a tensile fashion and when the current was lowered the stress would move toward a compressive value.

This phenomenon has been known for about 20 years but attempts to control the inherent deposit stress to such tight requirements are not generally attempted due to variations in the process at low levels within the plating run. The use of a commercially available monitor was used to measure the stress as plated on a membrane concurrent with the part plating. This allowed real-time measurements to be made and used for control. A computer control loop was established to achieve the task in accordance to a government owned patent (Martin Marietta Orlando Aerospace; assigned for government use). The computer was connected to a programmable 200 ampere Hewlett Packard (TM) power supply and commercial software was installed. The initial tests required that the current density be made more uniform in that the first mirror was slightly deformed and the thickness was not as uniform as was required along the length of the part. The commercial current density probe was modified to permit more precise measurements under the plating shields and toward the bottom of the mandrel. By readjusting the anodes and modifying the computer set-up to include a 12 bit rather than an eight bit card the control of both the stress and the deposit uniformity was excellent and produced mirrors of extremely good circularity.

6.0 FUTURE WORK

GOLD ADHESION TESTING

Subject: Gold to Nickel-Phosphorous Adhesion

Difficulty persists in achieving repeatable results for processing of the AXAF-S demonstration shell through the gold plating step. Low but uniform adhesion must be achieved with minimal surface degradation to the highly polished electroless nickel. After separation, the shell must not exhibit any form of material residue or staining. Evidence of material from the environment as well as material from the process has persisted at the interface on all four parts fabricated to date.

PRELIMINARY TEST PROCEDURE:

- 1.0 Prepare Ni-11% P samples for polarization studies.
 - 1.1 Plate coupons
 - 1.2 Plate load cell conical punch samples
 - 1.3 Polish
- 2.0 Scan polished Ni-P surface in 0.1N NaOH.
 - 2.1 Observe oxidation and oxide reduction using cyclic voltammetry.
 - 2.2 Add Ni-Rochelle Salt step - repeat.
 - 2.3 Compare Ligands of differing coordination
 - 2.3.1 Nickel-Rochelle Salt pH = 10 - 11
 - 2.3.2 Nickel-EDTA pH = 10 - 11
 - 2.3.3 Nickel Acetate pH = 10 - 11
 - 2.4 Compare higher valence cation $M^{+3,6}$ in place of Ni^{+2} .
 - 2.4.1 Aluminum Sulfate pH = 10 - 11
 - 2.4.2 Mo or W complex pH = 10 - 11
- 3.0 Based on results - eliminate further testing any point an unsuccessful continuous conductive film is achieved.
 - 3.1 Plate gold over passivation film
 - 3.2 Remove gold with cellophane tape
- 4.0 Plate conical adhesion samples with best processes determined.
- 5.0 Push samples apart with Load Frame in compression.
- 6.0 Produce process for test parts.

- 6.1 Plate coupons using best processes
- 6.2 Separate coupons and mirror replicate surface
- 7.0 Inspect using Interferrometry, AFM, ESCA and SEM.
- 8.0 Fabricate AXAF - S mirror per defined length process below.
 - 8.1 Plate
 - 8.2 LN₂ Separation
- 9.0 Inspect in visible and if good enough in X-ray bands.

PLATE PART TO DEFINED LENGTH TO AVOID CUT OFF OPERATIONS

MODIFIED EXISTING MANDREL EXPERIMENT:

- 1.0 Machine existing mandrel to accommodate shields.
 - 1.1 Design modifications
 - 1.2 Machine grooves
 - 1.3 Machine stop off pieces
 - 1.4 Assemble
- 2.0 Plate to test edge effects.
 - 2.1 Passivate in Rochelle salt solution (No Nickel)
 - 2.2 Plate gold
 - 2.3 Plate nickel using computer control

Note: Must remeasure effect of changes on current density.

- 3.0 Remove shell and inspect per above test.

PERFORM CLEANING TESTS ON X-RAY MIRROR SURFACE

CONTAMINATION CONTROL AND REMOVAL:

- 1.0 Test surface contamination removal using polar non-polar and aqueous solvents.
 - 1.1 Continue studies by Jeff Sanders ITTRI Inc.
SEM, ESCA and AUGER
 - 1.2 Measure effect of time, concentration and temperature
when using oxidants such as hydrogen peroxide.
- 2.0 Determine cleaning material requirements and specifications.
- 3.0 Determine clean room and environmental requirements.
- 4.0 Determine purge gas and rinse water requirements.

FIGURES:

Passivation Data;

Figure (1) shows the growth of the oxide film electrochemically. The peaks are the current response to a potential scan indicating the change in charge transfer associated with stoichiometry changes.

Figure (2) shows the growth of the oxide film in a complexing media containing nickel. Note that the process shows higher polarization and the peaks are much broader and that only two slow (rate determining) steps are observed indicating that the formation of a single oxide stoichiometry is more easily controlled.

Figure (3) is the same as Figure (2) except the nickel is not present.

Figure (4) represents the actual passivation behavior of the AXAF-S mandrel with a fixed applied current and the resulting increase of surface potential measured with a saturated calomel electrode.

ITTRI ESCA data;

Figures (5), (6) and (7) show the ratio of nickel, phosphorous and oxygen with different passivation conditions as measured by Electron Spectroscopy Chemical Analysis (ESCA).

NNR = No Nickel, Rochelle salt solution

FNR = Fresh Nickel containing Rochelle solution

CNR = Used Rochelle solution with colloidal nickel precipitate

No Passivation is sample exposed to air only after lapping

NaOH is pH 11 sodium hydroxide and no nickel or ligand

Note that the highest O/Ni and Ni/P is the NaOH alone indicating that the oxide proceeds to a higher oxygen stoichiometry as indicated in the electrochemical tests Fig. (1) and (2). The lowest oxygen to nickel is as expected the air exposed only sample.

Electroformed Nickel Process Data;

Figure (8) a and b are examples of the tracking of the current and the stress vs time. Figure (8) c is the stress versus the current density response over a test period of time.

This data shows the computer response to time dependent changes in the stress of the nickel while in real-time plating control.

Figure (9) a and b shows the current density variation measured with the eddy current probe. Note the improvement as variables were adjusted.

Figure (9) c shows the configuration of the nickel plating process for the low stress plating.

Laboratory and equipment;

Figures (10) and (11) show the laboratory layout as it exists.

Figures (12) - (14) show the proposed improved layout.

APPENDIX I

Fabrication Process Summary:

- 1) Heat treat forged aluminum alloy to zero stored energy
- 2) Machine to near net shape
- 3) Heat treat to remove machining stress
- 4) Single point diamond machine to net shape
- 5) Plate 11% phosphorous in nickel (Ni_3P) alloy by chemical plating
- 6) Heat treat nickel phosphorous alloy for improved adhesion
- 7) Single point diamond machine to final net figure and shape
- 8) Polish to final surface quality using MSFC procedures
- 9a) Form thin oxide film electrochemically
(Shells 1, 2, & 3);
- 10a) Plate gold from alkaline non-cyanide process 1 micron thick
- 9b) Allow to air oxidize rather than 9a
(Shell 4)
- 10b) Plate gold from commercial source using vacuum processing instead of electrolytic process 10a
- 11) Plate nickel to 1 millimeter thickness using stress controlled in real-time by proportional closed loop computer feedback from monitor
- 12) Clean part and cut off to length with cutting wheel on precision turning center
- 13) Clean part and chill with liquid nitrogen until separation at gold/electroless nickel phosphorous interface occurs
- 14) Final clean with solvent and if needed oxidant of hydrogen peroxide or ammonium persulfate - ammonium carbonate mixture to remove any residual foreign material
- 15) Inspect.

APPENDIX II.

Aluminum Heat Treatment Definitions

Aluminum stock purchased in the 2024-T6, T651, T6511 or T6510 condition will have been quenched from the solution temperature and precipitation aged. This process causes heavy sections of plate and bar to have a non-uniform internal stress due to stored energy related to the kinetic differential temperature in the stock during cooling and also due to complex metallurgical processes involving dislocation pinning by the Al-Cu and trace metal supersaturated inter-metallics in a kinetic fashion as the section is cooled non-uniformly.

This kinetic solidification process causes a distribution of stress such that the outer surfaces will typically be in compression and the inner surface in tension. The distribution for a three inch thick section is about -15 to +15 KSI obtained in cold water quenching and -5 to +5 KSI obtained in hot water or propylene glycol quenching. The lower stress process may also reduce the yield and ultimate strength values.

This stress can be reduced in the bulk metal by using a liquid nitrogen quench followed by a steam thermal shock prior to the precipitation hardening step. This process is patented as U.S. Patent 2,949,392 and while not well documented, appears to create a stable low stress condition primarily by grain boundary motion and to a lesser extent, various dislocation mechanisms. This process is followed by the conventional precipitation hardening.

Additional stabilization is also added to T6 condition aluminum by a mechanical strain elongation step of about 3% which is designated as T-6511. This step aligns the non-uniform structure in the direction of the strain and adds significantly to the fatigue and machining stability of critical components. This designation is for the mill and not typically for the user and would be very difficult to apply to this part.

The internal stress of bulk aluminum product can be reduced to near zero by proper annealing which is designated "O" temper and is effected by heating the aluminum to 650-750 deg. F for 1.0 hour and allowing to cool slowly in air. The creep resistance of material in this condition under sustained load would be very poor. This process reduces the strength and hardness due to extended precipitation of the Al-Cu and recrystallization grain growth and also increases the spacing of the dendritic Al-Cu pinning sites. Full annealing at 750 deg. F will leave the aluminum susceptible to very long term room temperature precipitation hardening which may induce slight stresses causing optically discernible distortion. In 2024 this process is not as severe as in other alloys but will still occur for as much as one year.

For the 2024 alloy the Al-Cu dissolution at 5% is commenced at 340 deg. F. This mandates that any heat treatment at this temperature or above after precipitation hardening will soften and reduce the strength of the metal albeit concurrently reducing stored energy and residual stress. At 350 deg. F the softening is gradual and occurs over approximately 3 weeks reducing the yield from about 40 KSI max to about 30 KSI. At higher temperatures the time for reaching a minimum yield and ultimate strength is reduced and the stress reduction is more significant. An annealing temperature of 500 deg. F will cause significant softening after about one hour and will reduce the yield and ultimate strength to nearly minimum after about 100 hours. Note that the properties of heavy pieces such as the mandrel will vary due to the original variance in properties described.

The material could be purchased as 2024-T4 which would then be heat treated back to the solution condition and subjected to the thermal shock conditions and finally precipitation hardened after rough machining. Machining of the T-4 temper is not usually performed since the material is somewhat "gummy". This however would produce a relatively uniform distribution of low residual stress.

When aluminum is purchased in the T-4 or T-451 condition the precipitation heat treatment consists only of the room temperature aging. Immediately after quenching the alloys are almost as ductile as fully annealed even though the copper is quenched into the bulk aluminum in solution. At room temperature the precipitation hardening process may continue for months. Often the material is to be drawn or worked in some way which requires ductility to be maximum. In this case the material can be ordered refrigerated (AQ) to slow the precipitation hardening process. The subsequent hardening after drawing or working can then be to the T-6 condition by the user. The internal stress of components fabricated in this manner is still significant and deformation can occur upon machining and heat treatment. The difference is that upon completion of all manufacturing steps the parts are in the strongest and hardest condition (T-6 or T-7) and have been stabilized after manufacturing.

The following original procedure was used to produce the first mandrel. Subsequent testing suggested lower times for the aging and stress relieving steps per NASA MSFC.

HEAT TREATMENT PROCESS FOR AL 2024 MANDREL

CLEAN WORK AREA OF ANY STEEL CHIPS WHICH COULD
CONTAMINATE ALUMINUM PART

1.0 Rough Machining

Machine forging to within 0.060 I.D. and O.D. final dimension.

1.1 The material is expected to be very soft and easily deformed ("gummy") causing severe surface deformation to about 1/2 the depth of a given cut.

1.2 The surface will be easily dented by tooling strikes or handling.

1.3 The surface cuts should be reduced in depth as the net shape is approached accordingly.

2.0 Heat-Treatment

2.1 Solution heat treatment

2.1.1 Fixture with the thermocouples inside and outside and at each end.

2.1.2 Soak times-air furnace per MIL-H-6088F Table IV

2.1.3 Temperature 910 deg. F min.
Temperature 930 deg. F max.

It is mandatory that the maximum not be exceeded in order to avoid exceeding the initial eutectic copper aluminum melting temperature.

2.1.4 Quenching

Normal quenching temperature for 2024 in water quench is 100 deg. F starting temperature with sufficient water to avoid exceeding 140-180 deg. F. For this application the "uphill" quench will be used and boiling water or steam is the preferred quench. The dwell time from furnace to quench must be minimal requiring removal of thermocouples in minimal time.

2.2 Precipitation Heat Treatment

Precipitation hardening and strengthening occurs after the solid solution quench above. This will be 18 hours at 375 deg. F +/- 5 deg. F to achieve T6 condition.

2.3 Stress Relief

2.4 Heat for 1.0 hour at 500 deg. F.

2.5 Air cool at 50 deg. F/hour until 350 deg. F is reached. Air quench beyond this point.

2.6 Reverse Quench Option (Time Permitting)

In order to fully stress relieve the part it is desirable to perform an additional cryogenic treatment as follows:

2.7 Immerse part in liquid N2 and allow to stabilize.

2.8 Steam blast within 7 seconds of removal from LN2 until at least 160 deg. F is attained in all sections.

2.9 Air Cool

3.0 Final Machine Prior to SPDT
(RECLEAN AREA IF STEEL CHIPS ARE PRESENT)

In order to minimize efforts on the single point diamond turning operations a near-net shape is required. The heat-treatment will have distorted the part. Return to machine after heat treatment.

3.1 Machine to net shape requirements determined by fixturing change accuracy (CA +0.010 on all dimensions typically). Note the contour requirements.

3.2 Cut with successively smaller feed depth until final cut is no more than 0.002 inch.

3.3 Clean part to avoid contamination.

3.4 Contact EB-23 R. Rood for shipping and handling instructions.

4.0 Post Machining Stabilization

4.1 Heat part to 200+/- 10 deg. F at 50 deg. F/Hour

4.2 Soak for 1.0 hour

4.3 Cool to 150 deg. F at 50 deg. F/hour (1 hour)

4.4 Cool part to room temperature uncontrolled

4.5 Cool part to -100 deg. F +/- 10 deg. F at 50 deg. F/hour

- 4.6 Soak for 1.0 hour.
- 4.7 Warm part to room temperature at 50 deg. F/hour maximum.
- 4.8 Return to EB-23 SPDT.
- 5.0 PERFORM MACHINING FOR ELECTROLESS NICKEL
- 6.0 PLATE ELECTROLESS NICKEL
- 6.1 Post Electroless Nickel Stabilization
- 6.2 After plating Electroless Nickel heat part to 320 deg.
F +/- 5 deg. F at 100 deg. F/hour.
- 6.3 Soak for 4.0 hours +/- 10 min.
- 6.4 Cool to 150 deg. F at 50 deg. F/hour max.
- 6.5 Air cool to room temperature.

APPENDIX III

PLATING PROCEDURES:

OPERATING INSTRUCTIONS FOR AXAF - S PLATING

STAPLETON ELECTROLESS NICKEL PROCESS:

AUTOBOND 215, CLEANER

MAKE UP	215A	10% Volume
TEMP F DEGREE		150-170
TIME MIN.		2-5

AUTOBOND 245, DEOX

MAKE UP	245A	10% Volume
TEMP F DEGREE		55-75
TIME MIN.		2-5

AUTOBOND 361, ZINCATE

MAKE UP	360A	25% Volume
TEMP F DEGREE		55-75
TIME SEC.		60-90

AUTOBOND 880, NICKEL STRIKE

MAKE-UP	380A	100% Volume
TEMP F DEGREE		55-75
PH		6.2-6.4
ASF		25
TIME MIN.		5

AUTONIC HX, ELECTROLESS NICKEL PLATING

MAKE-UP 50 GALLONS

AUTONIC HXPA	15%	7.5 GAL.
AUTONIC LNS	5%	2.5 GAL.
DISTILLED WATER	80%	40 GAL.

CONDITIONS

NICKEL	6.65 G/L
HYPOPHOSPHITE	35 G/L
TEMP	185 DEG. F
pH	4.2

LOWER pH WITH SULFURIC ACID.

CAUTION: DO NOT HEAT IN EXCESS OF 195 DEG. F OR THE SOLUTION MAY SPONTANEOUSLY DECOMPOSE.

PROCEDURE FOR NICKEL REPLENISHING PROCESS:

1. TITRATE NICKEL
2. MAKE ADDS TO PROCESS
3. CHECK pH AND TEMPERATURE, ADJUST AS REQUIRED.
4. RECORD RESULTS IN LOG.

PASSIVATION PROCESS FOR GOLD RELEASE:

NOTE: SUBJECT TO CHANGE BASED ON EXPERIMENTS BEING CONDUCTED

SODIUM HYDROXIDE 8.0 G/L

ROOM TEMPERATURE
18-8 SS ANODES
D.I. WATER

1. CATHODIC IN ABOVE AT 20 ASF

TIME SEC. 30.0

2. NICKEL ROCHELLE SALT

NICKEL SULFATE HEPTAHYDRATE	2.0
ROCHELLE SALT	28.0
SULFURIC ACID	1.0
SODIUM HYDROXIDE	pH 11.0

PASSIVATE ANODICALLY IN ROCHELLE
SALT SOLUTION AS FOLLOWS:

Monitor both current and potential vs SCE against time

Raise current manually at about 20 - 30 mA/Ft square

Observe reference potential slope to achieve 18 seconds to
passivation potential

Hold at passivation potential for 6 - 8 seconds

Shut off current and observe potential decay to assure that
the surface was not overpassivated.

Remove part and rinse thoroughly and place in gold with no
applied current.

TECHNI-GOLD 25 NON-CYANIDE GOLD PLATING:

<u>EQUIPMENT:</u>	TANKS	- FIBERGLAS, GLASS, POLYETHYLENE, PROPYLENE, PLASTIC LINED STEEL.
	ANODES	- PLATINUM PLATED TITANIUM OR TYPE 316 STAINLESS STEEL.
	AGITATION	- REQUIRED - RAPID

FORMULATION: READY TO USE

OPERATING CONDITIONS:

pH (ELECTROMETRIC)	- 9-10 (SEE NOTE 1)
SPECIFIC GRAVITY	- 9-10 DEG. BAUME' (SEE NOTE 2)
ANODE: CATHODE RATIO	- 2:1 OR GREATER
AGITATION	- RAPID
TEMPERATURE	- 120 DEG F (RANGE 100-150 DEG.F)
CURRENT DENSITY	- 1-5 ASF (OPTIMUM 3 ASF)
TIME TO PLATE 0.00005"	- 6.0 MINUTES AT 3 ASF

GOLD REPLENISHMENT:

EVERY 4 AMP-HOURS ADD 1.0 OZ. TROY GOLD AS TECHNI-GOLD 25 AND 0.35 GRAMS OF TECHNI-GOLD 25 BRIGHTNER POWDER. (SEE NOTE 3)

NOTE 1. pH ADJUSTMENTS SHOULD BE MADE USING A 20% SOLUTION OF SODIUM HYDROXIDE. AT NO TIME SHOULD THE pH BE ALLOWED TO DROP BELOW 8.0 OR ELSE DECOMPOSITION OF THE BATH WILL TAKE PLACE.

NOTE 2. SPECIFIC GRAVITY SHOULD BE 9 DEG. BAUME' (MINIMUM) BUT CAN RISE AS HIGH AS 35 DEG.

NOTE 3. IF PLATES ARE HAZY, SMALL AMOUNTS OF BRIGHTNER MAY BE ADDED UNTIL PLATE IS BRIGHT.

NOTE: BEWARE OF EXCESSIVE PASSIVATION AS CAUSE OF POOR PLATING.

ELECTROLYTIC LOW STRESS NICKEL PLATING PROCESS:

NICKEL METAL (SNR-24 CONCENTRATE) FOR 100 GALLONS (379 LITERS)	76.5 Gm/L 42% Volume 42 GALLONS
BORIC ACID FOR 100 GALLONS	30.0 Gm/L 25.0 POUNDS (11.37 Kg)
ADDITIVE "B" BROMIDE FOR 100 GALLONS	30.0 ml/L 100.0 OUNCES LIQUID (0.78 GALLONS) (2.95 LITERS)
pH ELECTROMETRIC ("SNAC" SULFAMIC ACID TO LOWER)	4.0
SODIUM LAUREL SULFATE (SNAP) 30 DYNE CM TENSION (10 - 12 SECONDS "SNAP TIME" ON 3 INCH SS RING) ADD IN SMALL INCREMENTS AND CHECK FREQUENTLY ON MAKE-UP START WITH 0.1 Gm/L	
PLATE ONE OR MORE SQUARE FOOT PANEL AT 5 ASF FOR 12 HOURS OR MORE TO REMOVE TRACE IMPURITIES	

COMPLETE PLATING PROCESS:

- 1.0 ALUMINUM ACTIVATION:
- 1.1 CLEAN PARTS WITH APPROPRIATE SOLVENT
(acetone or freon 113)
- 1.2 CLEAN PARTS USING AUTOBOND 215 PROCESS
2 MINUTES
- 1.3 RINSE THOROUGHLY USING D.I. WATER (2.0 Meg Ohm)
- 1.4 REMOVE OXIDE USING AUTOBOND 245 PROCESS
2 MINUTES
- 1.5 RINSE USING SEPARATE RINSE
- 1.6 ZINCATE IMMERSION COAT USING AUTOBOND 361 PROCESS
1 to 1.5 MINUTES
- 1.7 RINSE USING SEPARATE RINSE
- 1.8 REPEAT 1.4 THROUGH 1.7

- 1.9 PLATE THIN STRIKE COATING USING AUTOBOND 380 PROCESS
5 MINUTES
- 1.10 RINSE THOROUGHLY USING SEPARATE RINSE
- 2.0 ELECTROLESS NICKEL PLATING:
- 2.1 PLATE USING AUTONIC HX HIGH PURITY PROCESS
- 2.2 PLATE AT 185 DEG. F
- 2.3 PLATING RATE WILL BE 0.0004 INCH/HOUR
- 3.0 RETURN PART TO EB-23 FOR POST MACHINING/POLISHING
- 4.0 PASSIVATE NICKEL SURFACE:
- 4.1 IMMERSE IN SODIUM HYDROXIDE AS ABOVE
- 4.2 APPLY 20 AMPERES PER SQUARE FOOT CATHODIC TO PART
- 4.3 REMOVE CURRENT AND RINSE THOROUGHLY IN D.I. WATER - .
- 4.4 PASSIVATE AS OUTLINED
- 4.5 RINSE THOROUGHLY AND IMMERSE IN GOLD WITH NO CURRENT
- 5.0 GOLD PLATE AS ABOVE FOR 6 MINUTES
- 6.0 LOW STRESS ELECTROLYTIC PLATING PROCEDURE:
- ALLIED KELITE BARRETT SULFAMATE PROCESS
- 6.1 USE COMPUTER CONTROL FOR STRESS.
- 6.2 PLATE 0.040 INCH

0.1 M NaOH R.T.

Model 352 Corrosion Analysis Software, v. 1.00

DATE RUN: 03-11-93 TIME RUN: 12:59:14

CP -0.800 vs. R CT 60

SI 2.000E-03 SR 5.000E-03

FL NONE RT HIGH STABILITY

IT PASS ITA 1.000E+00

IP -0.800 vs. R

ST 4.000E-01

REF 0.24150 SCE

EW 0.000E+00

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NP 1573

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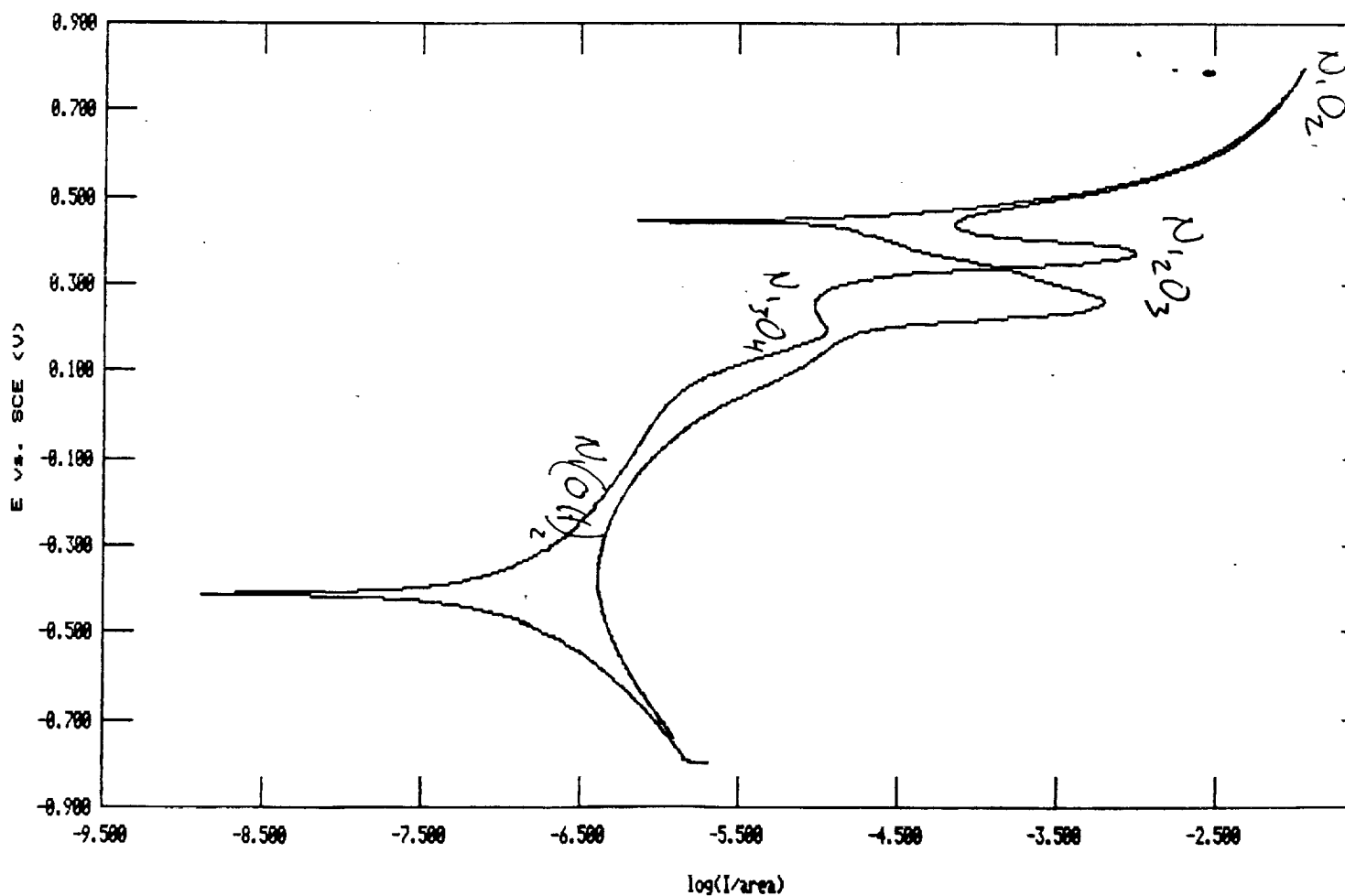
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FILE STATUS: NORMAL

FP 0.000 vs. OC

IR NONE

LS NO



F₁(1)

First scan 0.1M Rochelle Salt
2.1M Vit+

Model 352 Corrosion Analysis Software, v. 1.00

CP CYCLIC POLARIZATION

FILE STATUS: NORMAL

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CT 60

IP -1.000 vs. R

ID PASS

V1 1.000 vs. R

FP 0.000 vs. OC

SI 2.000E-03

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RT HIGH STABILITY

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LS NO

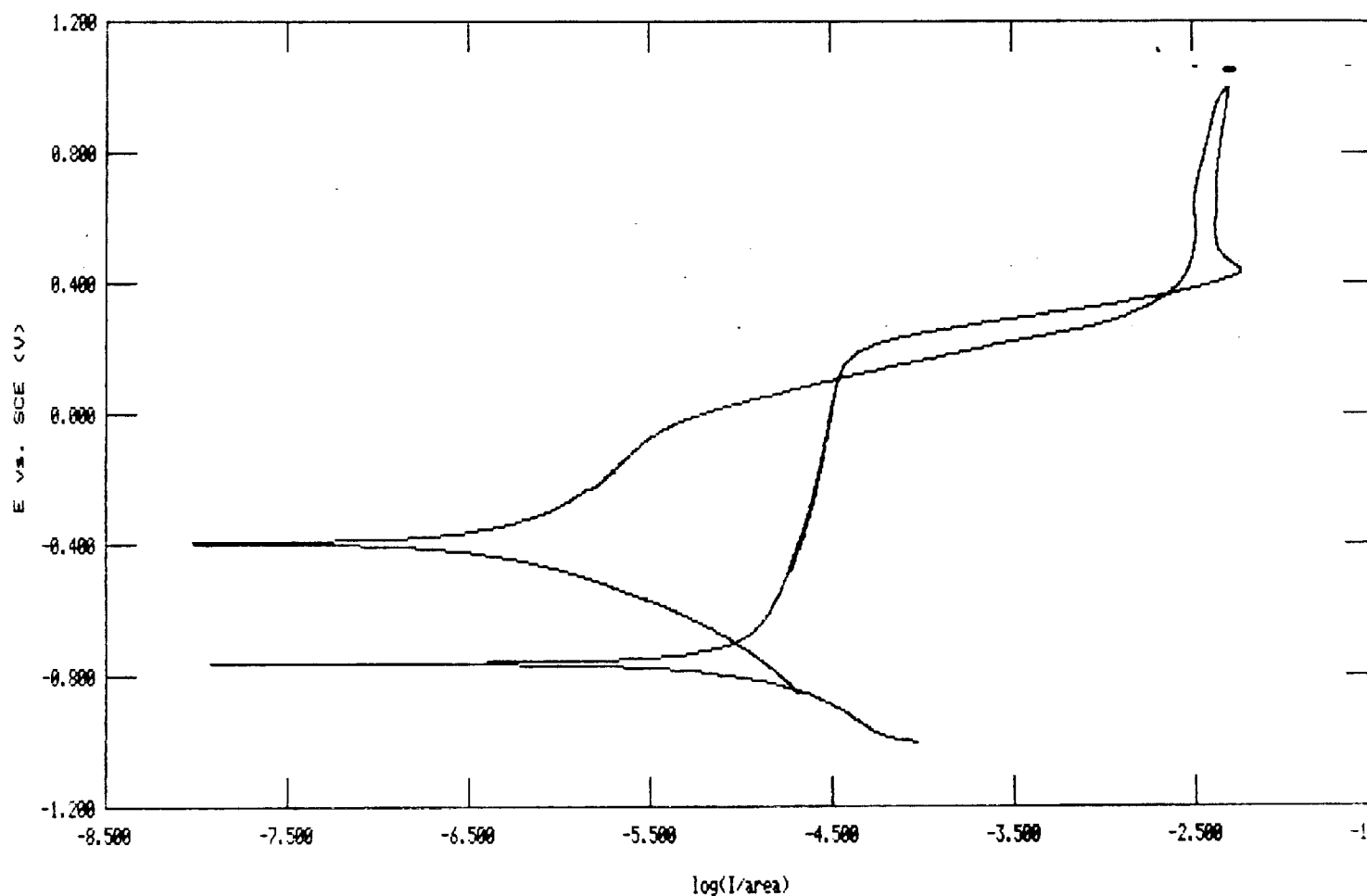
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DEN 0.000E+00

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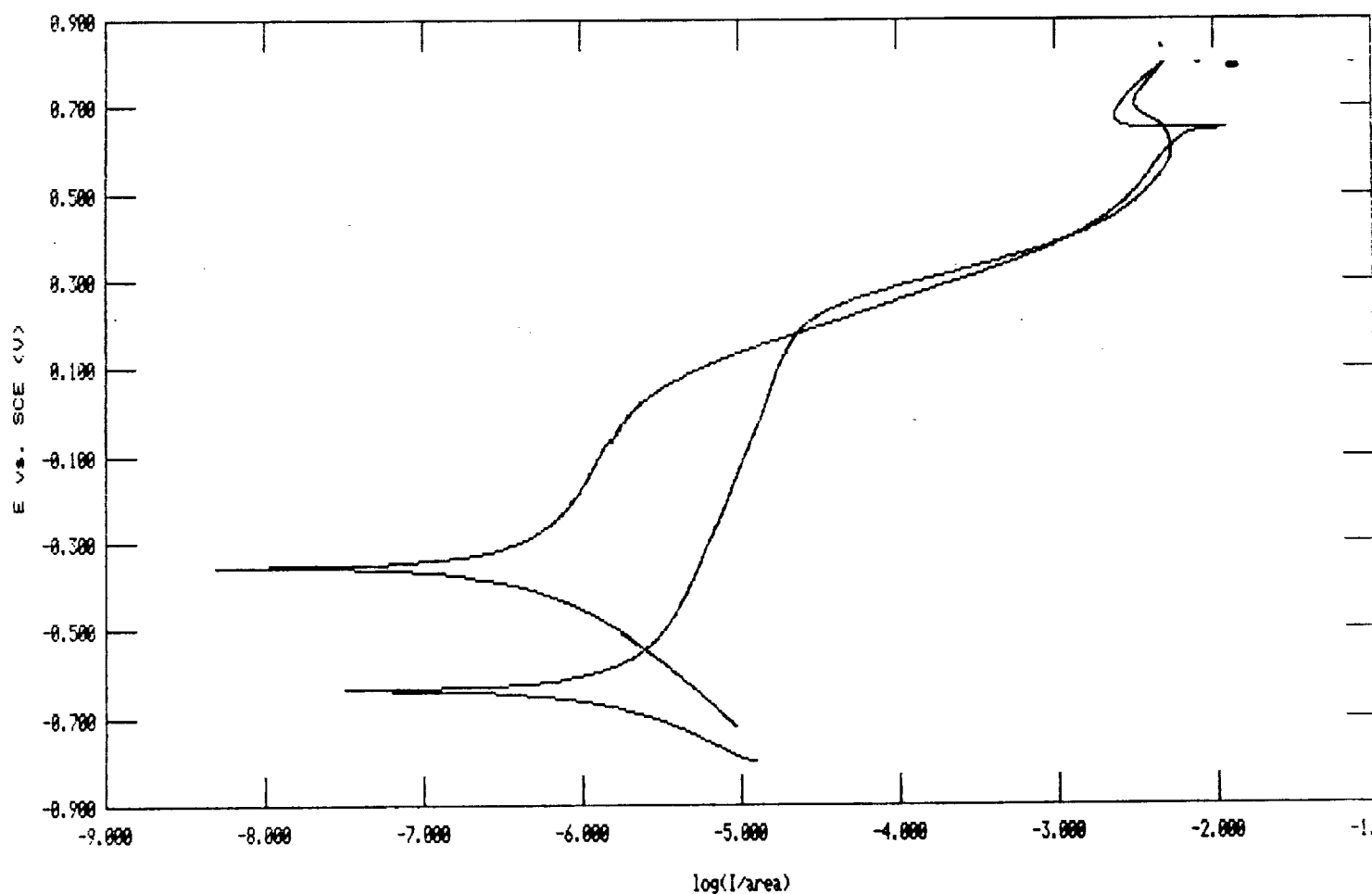
Fig (2)

N: 117₃P

R.T.

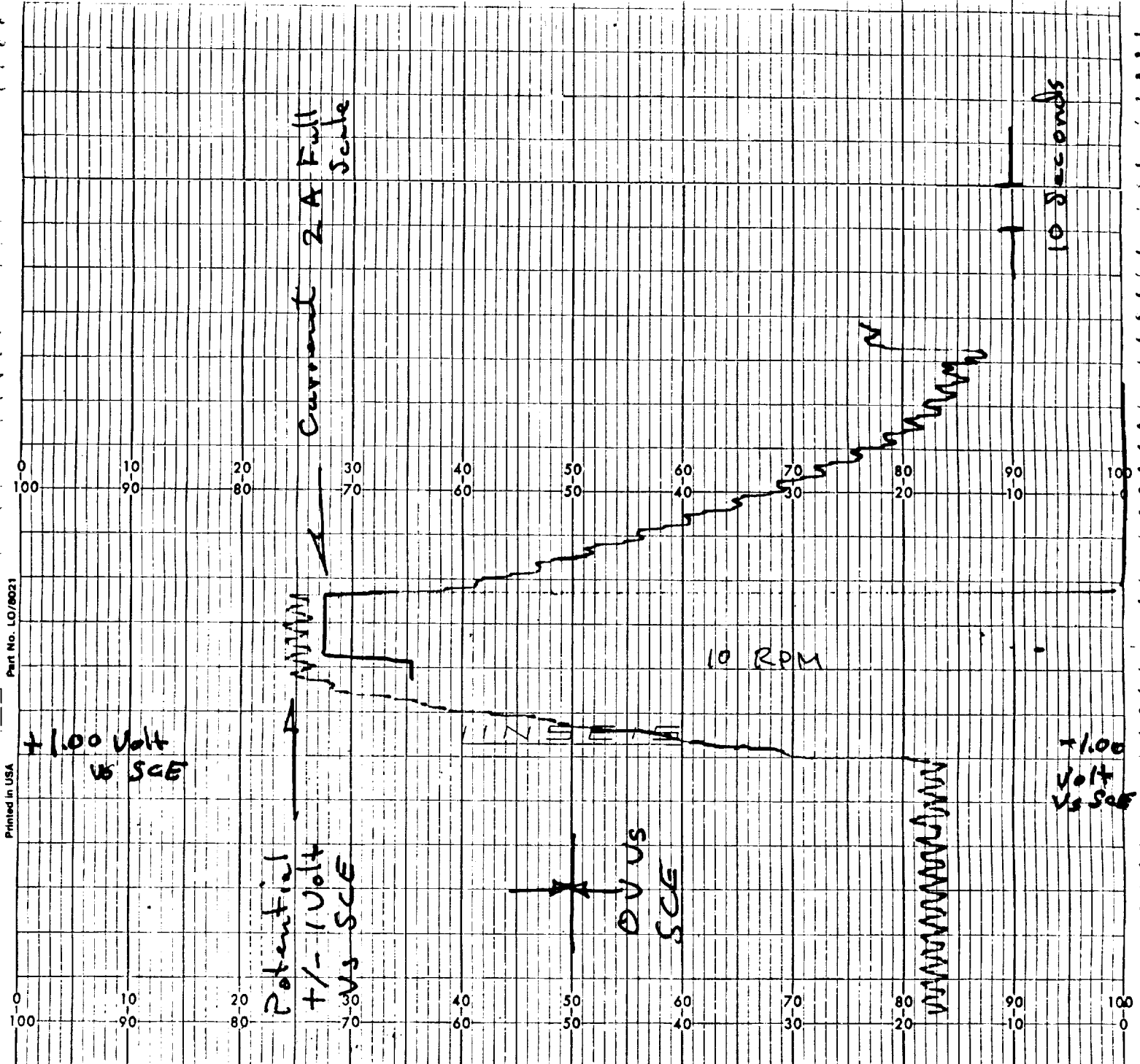
0.1 N NaOH
+ 0.1 M K₂Cr₂O₇ in

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SI 2.000E-03	SR 5.000E-03	ST 4.000E-01	CR AUTO	NP 1561	IR NONE
FL NONE	RT HIGH STABILITY	REF 0.24150 SCE	WRK SOLID	AR 1.000E+00	LS NO
IT PASS	ITA 1.000E+00	EW 0.000E+00	DEN 0.000E+00	OC -0.719	



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Fig (3)



AXAF-S Mandrel #2
June 25, 1993

Passivation
Potential & Current
vs
Time

Fig (4)

30 Seconds Passivation

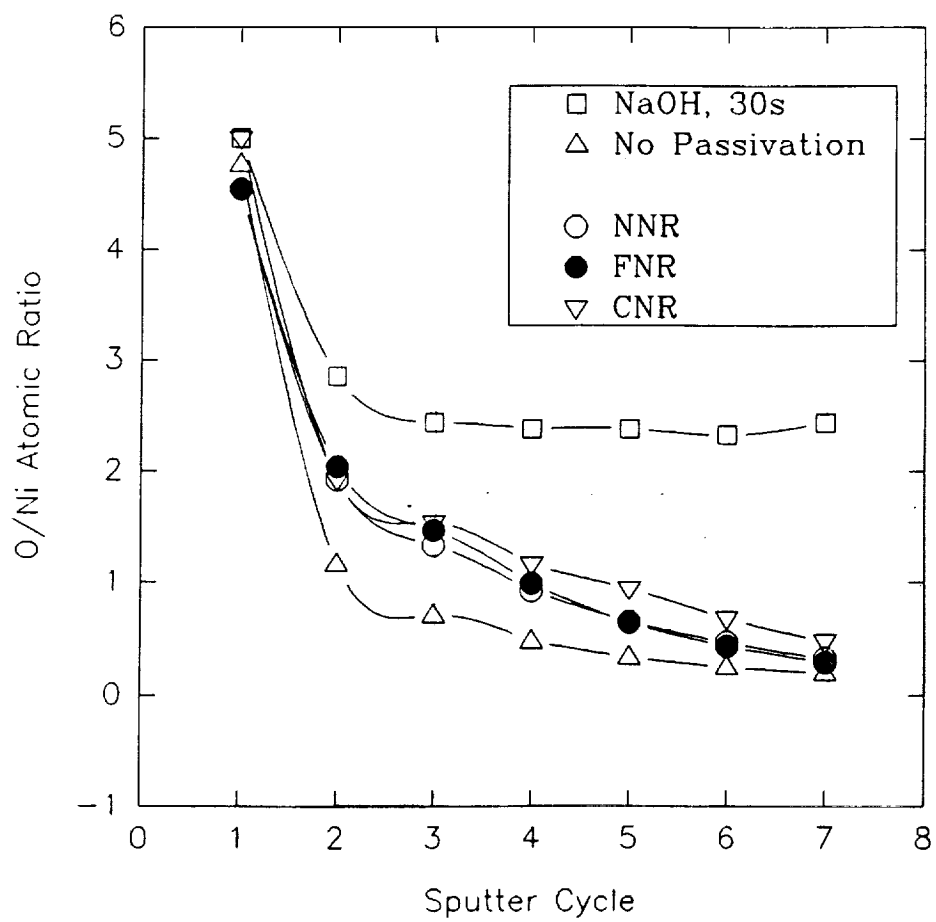


Fig (5)

30 Seconds Passivation

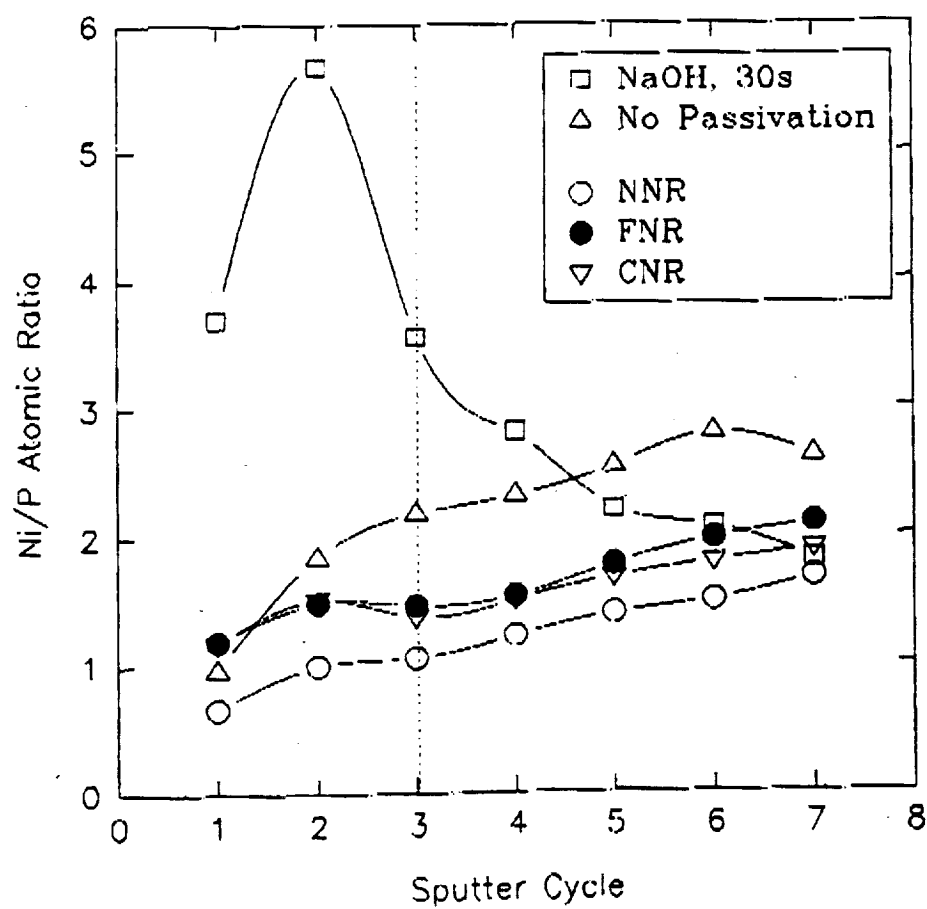


Fig (6)

30 SECONDS PASSIVATION

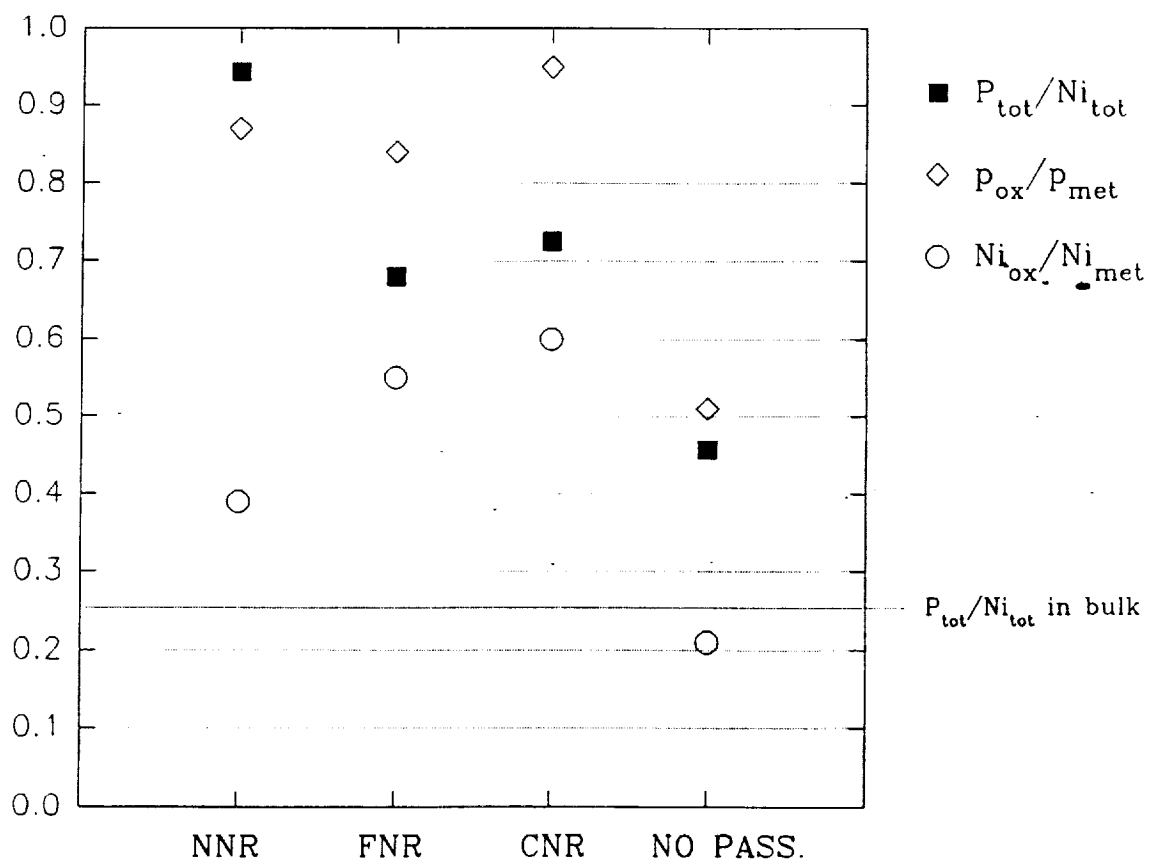


Fig (7)

AXAF S PART NUMBER 02 PLATING PROFILE

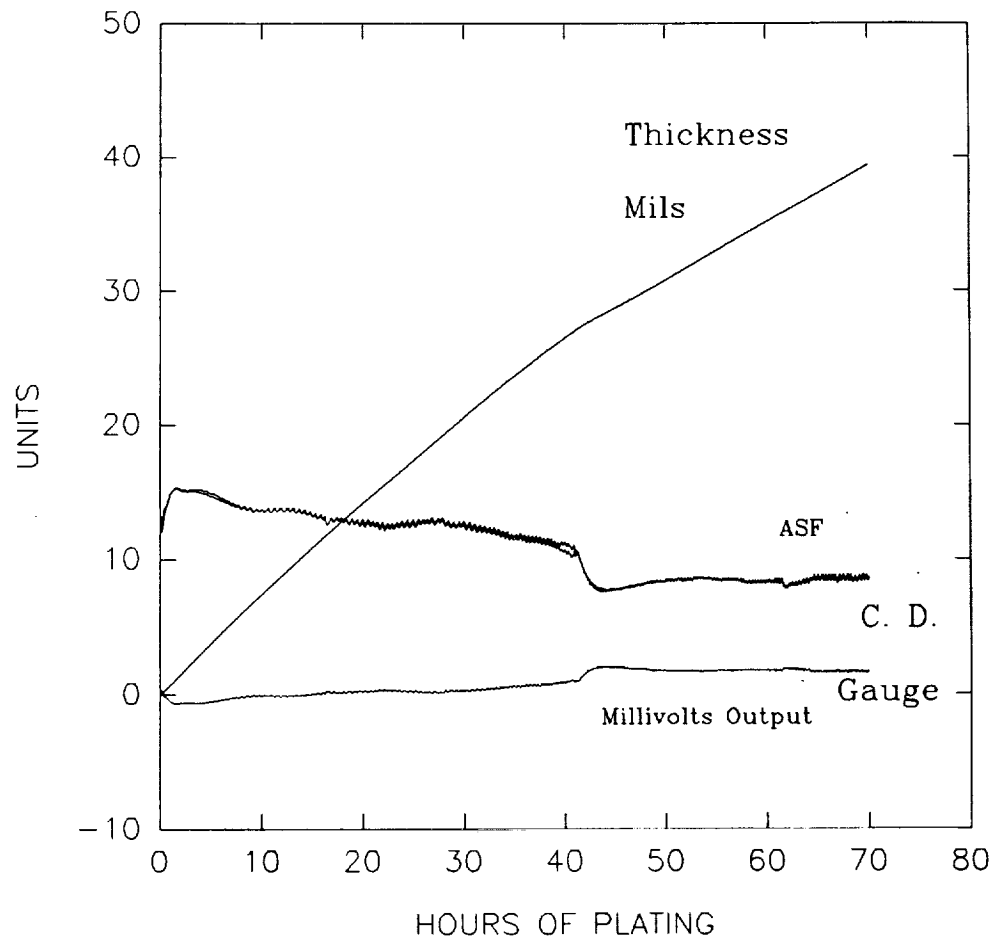


Fig. (8)(a)

AXAF - S MIRROR 2 FROM MANDREL 2

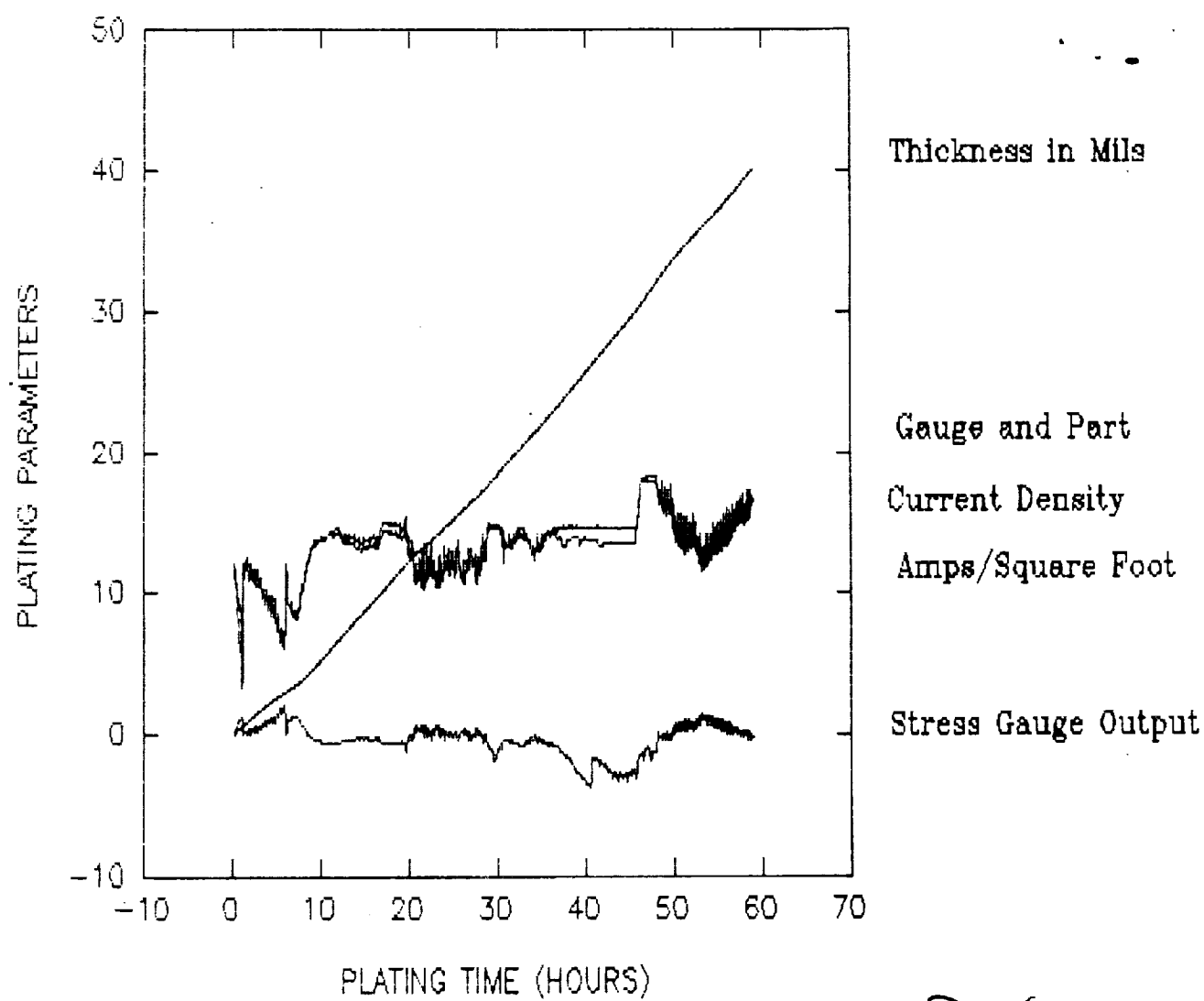


Fig.(8)(b)

LOW STRESS NICKEL PLATING

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 Rank 1 Eqn 6306 $y=a+b\ln x+c(\ln x)^2+d(\ln x)^3+e(\ln x)^4+f(\ln x)^5+g(\ln x)^6+i(\ln x)^8$
 $a=-4636.6825$ $b=-49.709112$ $c=107.76609$ $d=168.35699$ $e=130.96864$
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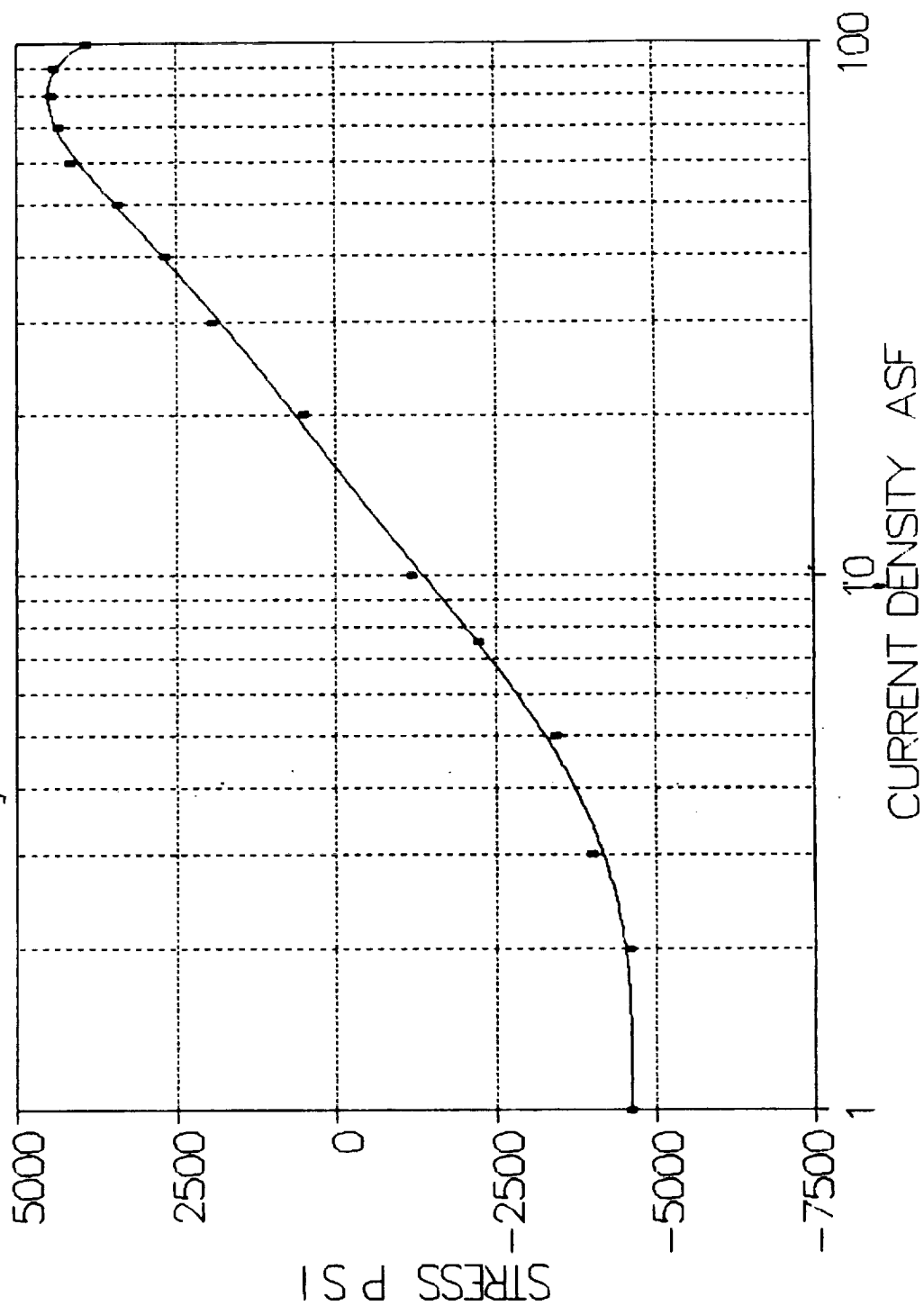
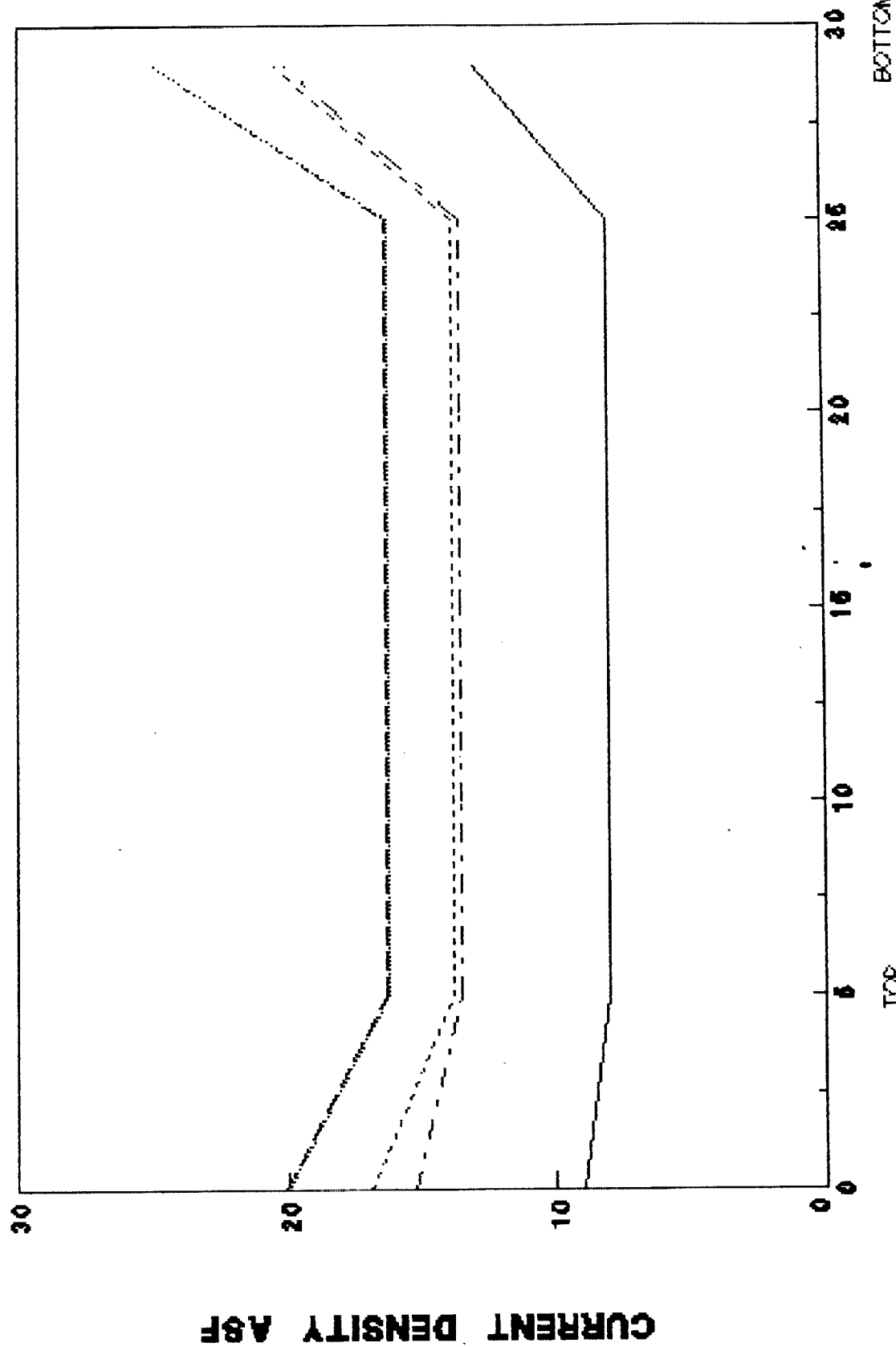


Fig (8)(c)

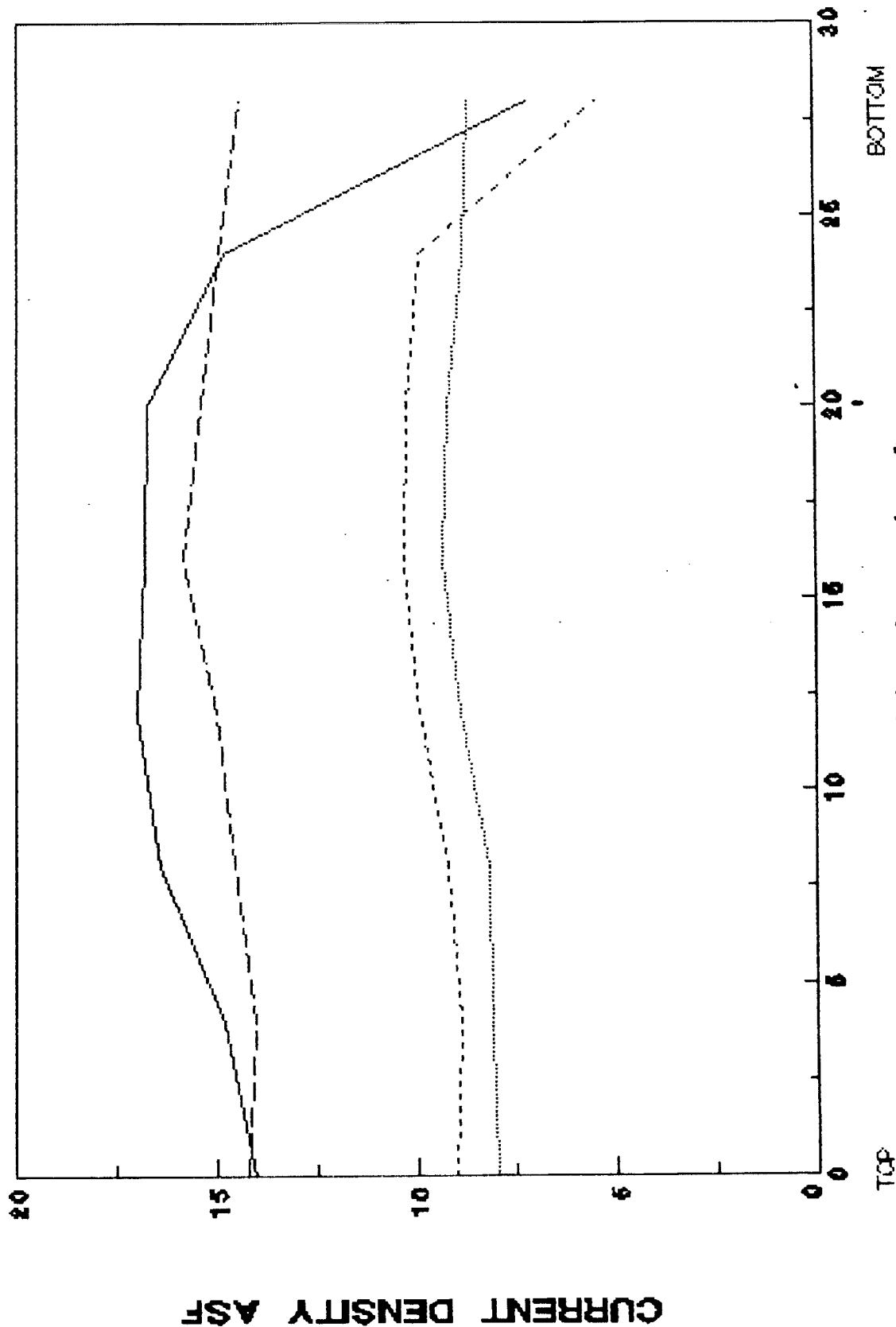
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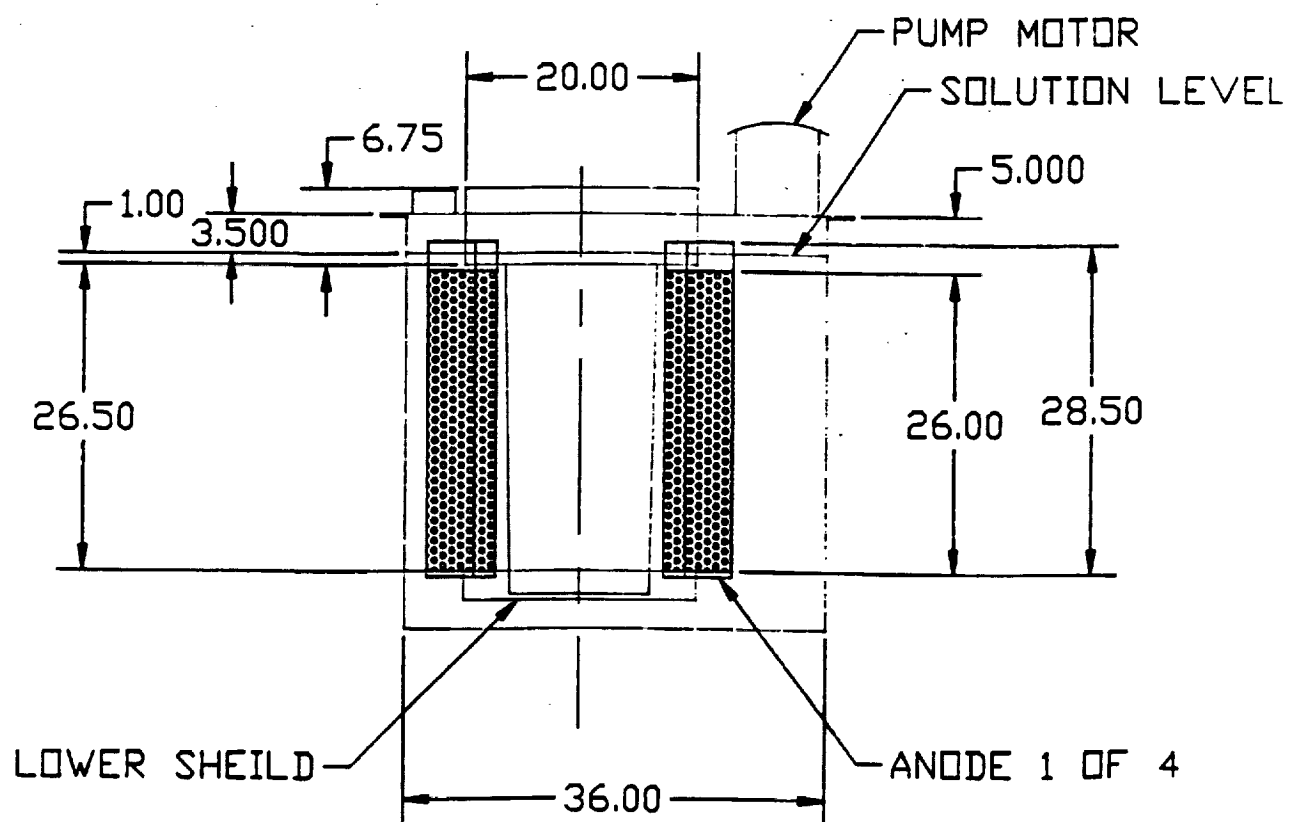
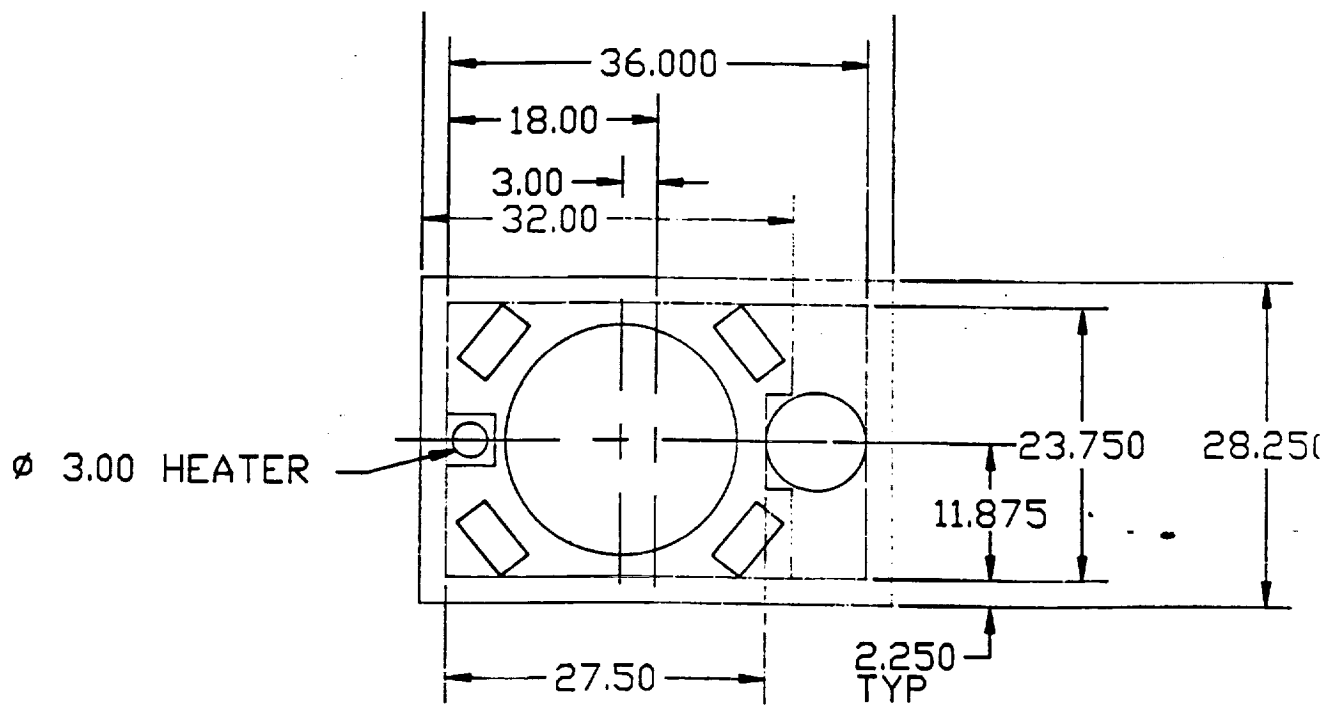
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 10 R 10 R 10 R 10 R 0 R



AXAF-S CURRENT DISTRIBUTION

— 100 AMP 1ST MOD - - - 80 AMP 1ST MOD ····· 100 AMP 2ND MOD - - - 80 AMP 2ND MOD





CAD/UAH
6-25-93

Fig (9) (c)

MATERIALS PROTOTYPE PLATING LABORATORY STEP I PLATING

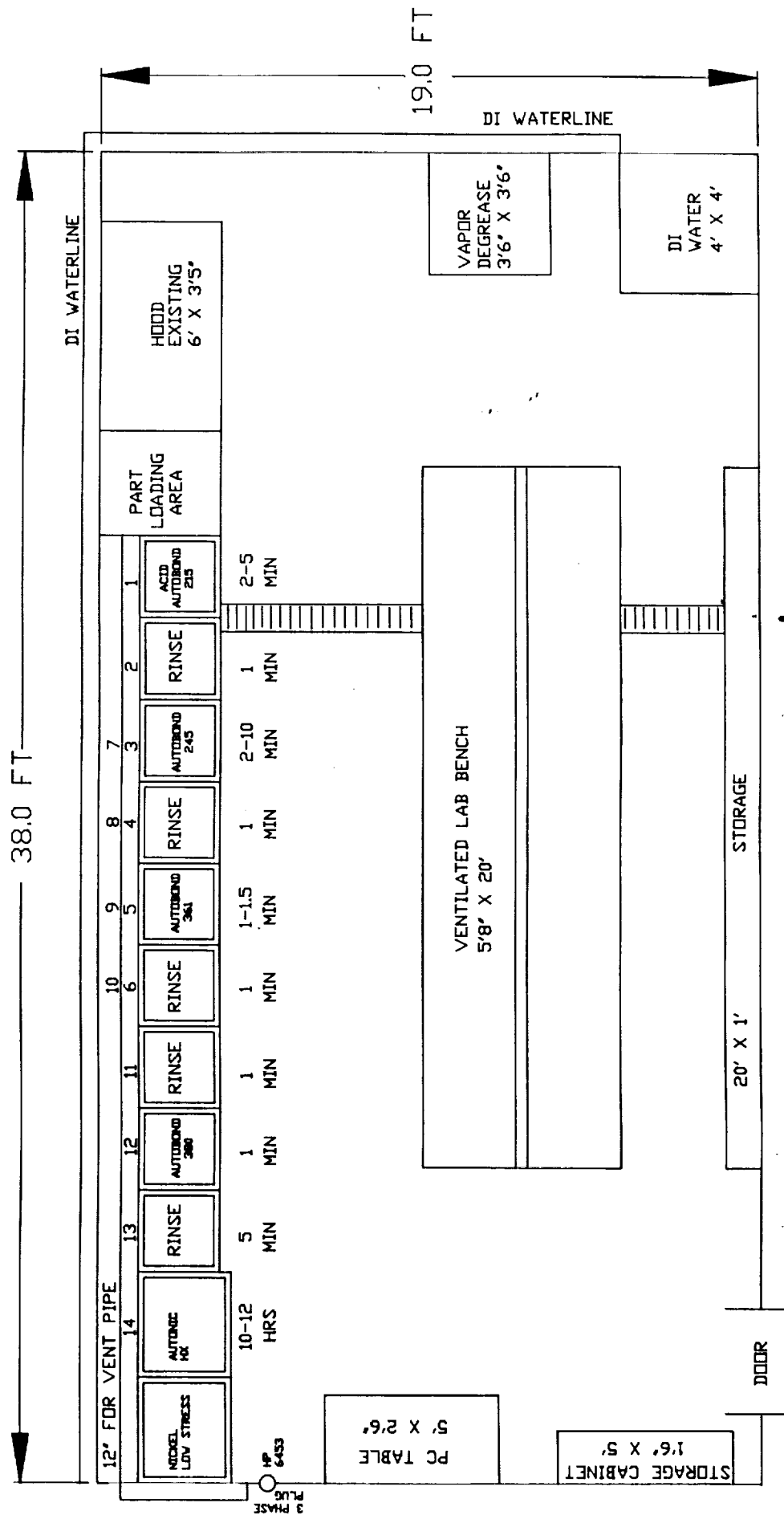


Fig-(10)

MATERIALS PROTOTYPE PLATING LABORATORY STEP II PLATING

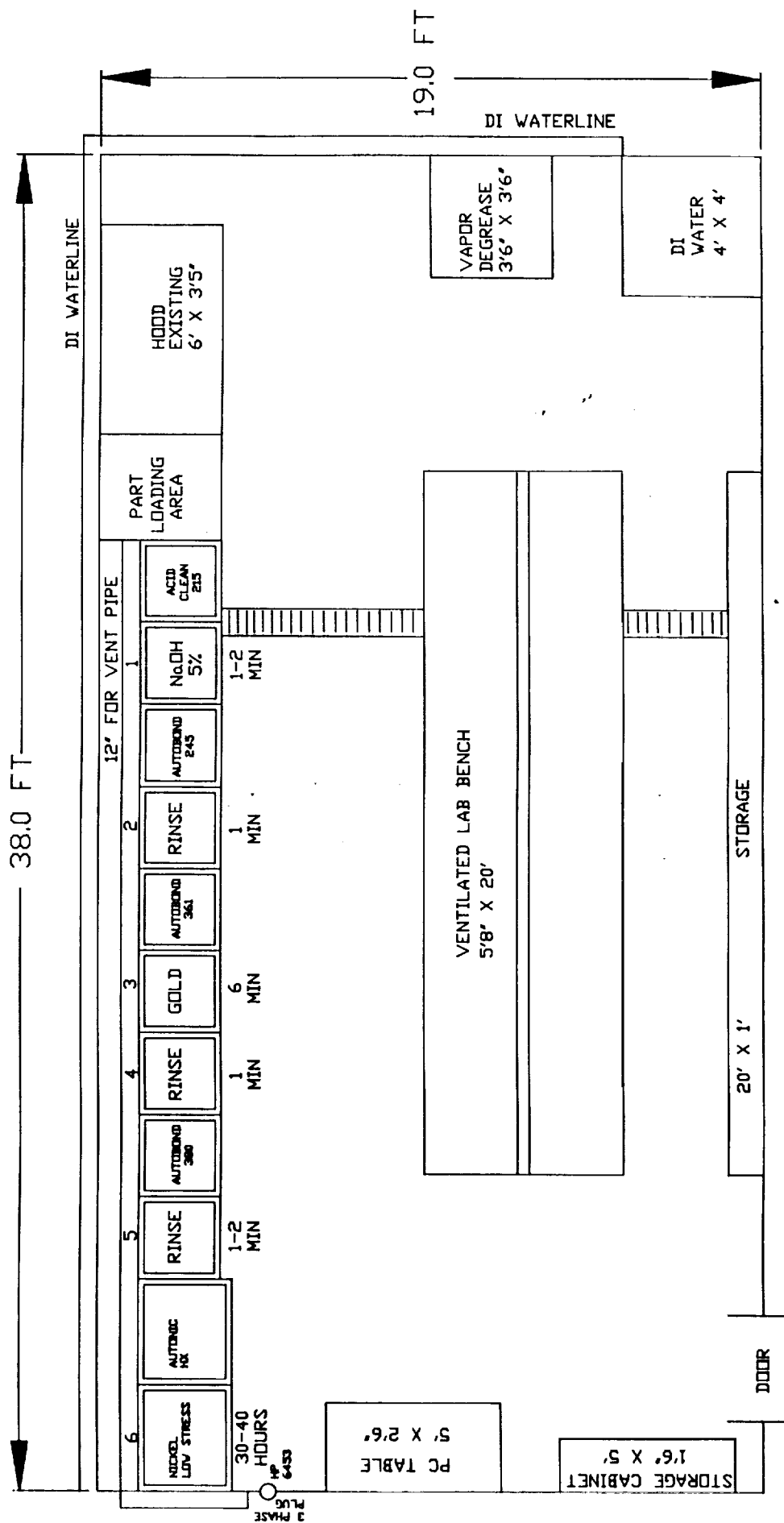
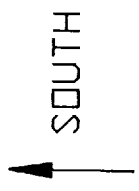
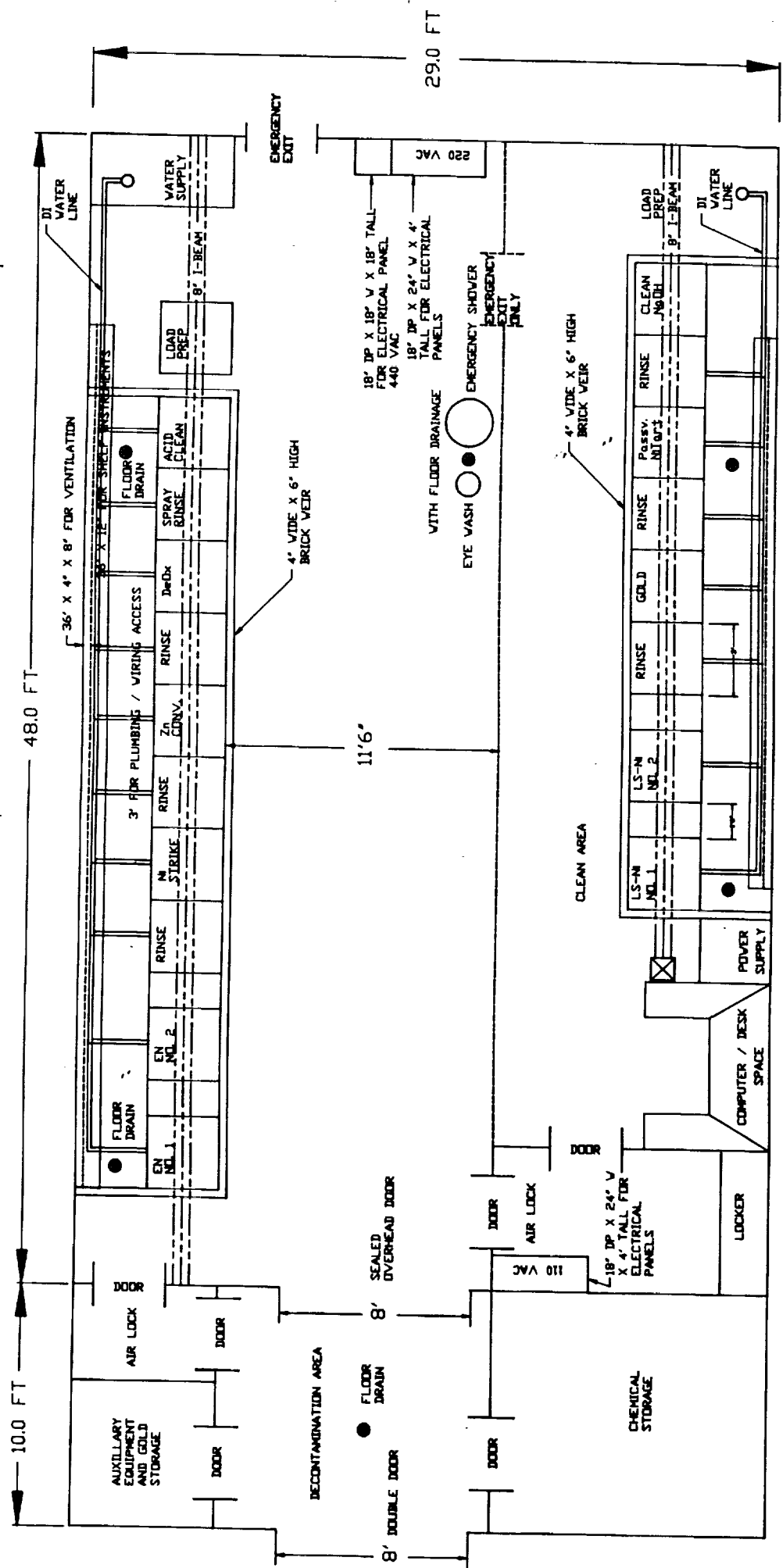


Fig. (11)



NEW AXAF PLATING LABORATORY



PLAN VIEW

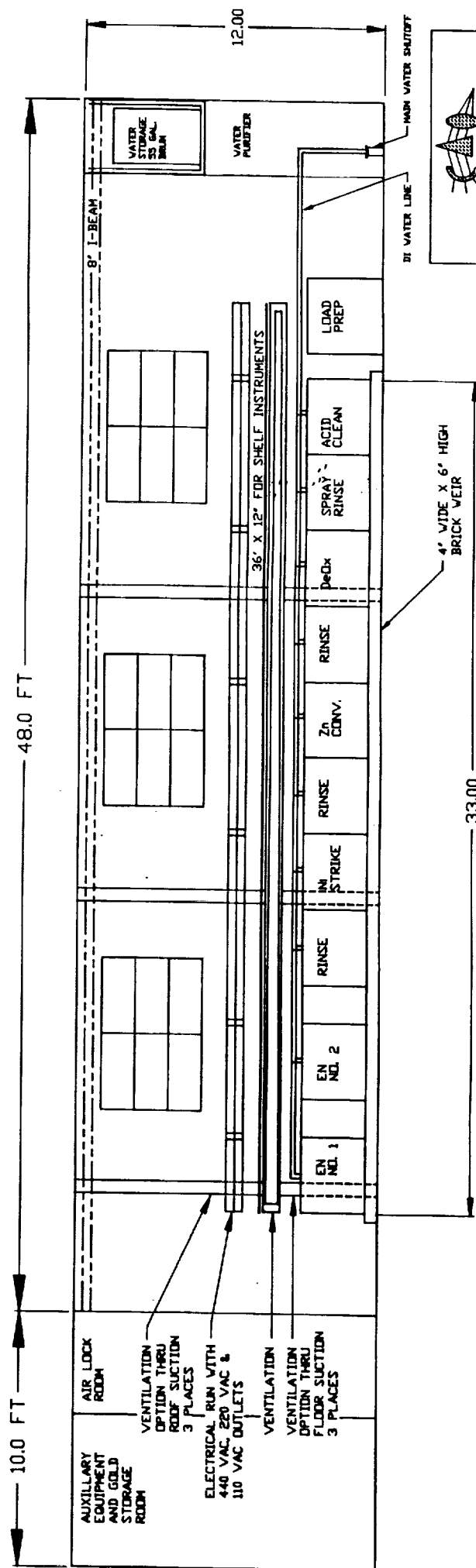
Center for Applied Optics
University of Alabama in Huntsville
Huntsville, AL 35899

Dr. B. L. Lefebvre
/ R. S. Soley
CONTRACT: H9550-00-0000-0001
PLATTED: SEPT 27, 1990

Fig (12)

NEW AXAF PLATING LABORATORY

SOUTH



Center for Applied Optics
University of Alabama in Huntsville
Huntsville, AL 35899

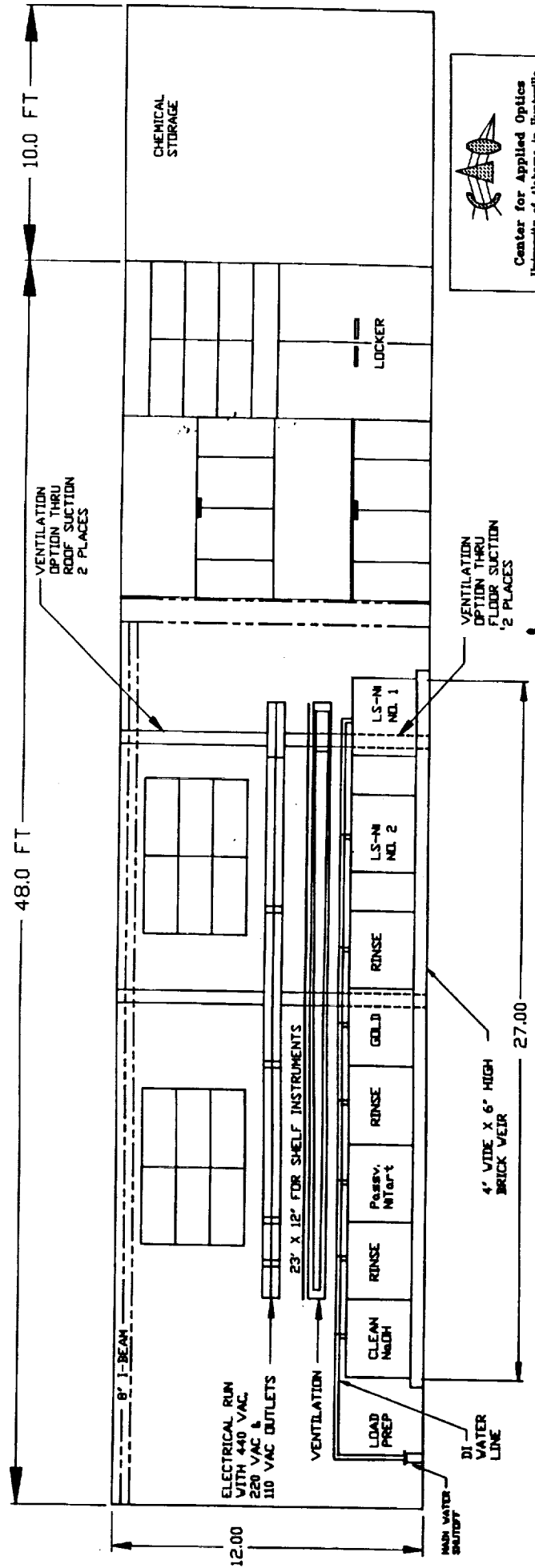
Dr. D. Capenham
/ Dr. B. Bailey
CONTRACT WASH-3869 D.O. 63
PLOTTED SEPT 27, 1993

ELECTROLESS NICKEL PLATING LINE

Fig. (13)

NEW AXAF PLATING LABORATORY

NORTH



Center for Applied Optics
University of Alabama in Huntsville
Huntsville, AL 35899

BY: D. Engelhardt
DATE: 10/1/73
CONTRACT: NAS-30496 R.O. 43
PLOTTED SEPT 27, 1973

ELECTROLYTIC NICKEL PLATING LINE

Fig. (14)

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